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**GEORGIA INSTITUTE OF TECHNOLOGY**  
OFFICE OF RESEARCH ADMINISTRATION  
**RESEARCH PROJECT INITIATION**

Date: February 19, 1974

Project Title: **Development of a High Stability Long-Life Field Emission Electron Source with a High Brightness**

Project No.: **E-18-607 and E-21-638**

Principal Investigator **Dr. A. T. Chapman & Dr. R. K. Feeney**

Sponsor: **CBS Laboratories (Division); Columbia Broadcasting System, Inc., Stamford, Connecticut**

Agreement Period: From 11/29/73 Until 8/31/74

Type Agreement: **Purchase Order No. 52501 (Fixed-Price); subcontract under F08606-73-C-0028**

Amount: **\$40,000**

Reports Required: **Program Schedule & Milestone List, Monthly Letter Reports, Phase II Topical Report, Abstract of New Technology**

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Defense Priority Rating: **DO-C9E under IMS Reg. 1.**

Assigned to: Schools of Ceramic Engineering and Electrical Engineering

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GEORGIA INSTITUTE OF TECHNOLOGY  
OFFICE OF CONTRACT ADMINISTRATION  
SPONSORED PROJECT TERMINATION

Posted  
6/28/77  
CH

Date: June 20, 1977

Project Title: "Development of a High Stability Long-Life Field Emission Electron Source with a High Brightness."

Project No: E-18-607/E-21-638

Project Director: Dr. At. T. Chapman, Dr. R. K. Feeney

Sponsor: CBS Laboratories (Division); Columbia Broadcasting System, Inc.

Effective Termination Date: 8/30/75

Clearance of Accounting Charges: All have cleared

Grant/Contract Closeout Actions Remaining:

- ☐ Final Invoice and Closing Documents
- ☐ Final Fiscal Report
- ☒ Final Report of Inventions
- ☒ Govt. Property Inventory & Related Certificate
- ☐ Classified Material Certificate
- ☐ Other \_\_\_\_\_

Assigned to: Ceramic Engineering/Electrical Engineering (School/Laboratory)

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E-18-607/C-21-658

MONTHLY TECHNICAL STATUS REPORT

1 January - 31 January 1974

HIGH STABILITY LONG LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332



# MONTHLY TECHNICAL STATUS REPORT

on

DEVELOPMENT OF A HIGH STABILITY LONG-LIFE EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This is the first Status Report, which covers the period from 1 January through 1 February 1974. The report is divided into several sections each covering a different aspect of the work.

## I. Preparation of Arrays Having a Small Number of Pins.

Mechanical shaping of a single cell was the first technique employed in the production of a small numbered pin array. A  $\text{UO}_2$ -W sample, initially brazed to a Mo stub, was ground to a sharp point with a diamond burr under a binocular microscope. The tip was delicately polished to achieve a flat surface containing approximately 500 pins. Subsequent chemical etching was employed to form exposed pins  $0.30\text{ }\mu\text{m}$  in diameter and about  $10\text{ }\mu\text{m}$  long with blunt tips. This geometry served as the initial attempt to characterize the field emission behavior from a small number of pins.

## II. Techniques for Selective Etching of $\text{UO}_2$ -W Composites.

It has been noted that  $\text{UO}_2$ -W composites will not dissolve in an etch of chromic, hydrofluoric, acetic, and nitric acids when the composite is copper coated on the back side. This effect will be used to produce a composite composed of a great many small isolated areas each containing a few pins. The emitter waver back surface will be coated with many small "islands" of copper and then etched. Etching will occur only in those areas where there



is a lack of copper, thus producing the desired configuration. It is believed that samples with dimensions as small as  $5\text{-}10\text{ }\mu\text{m}$  in diameter can be produced in this manner.

### III. Low Voltage Field Emission Arrays.

Work is in progress to fabricate a structure capable of low voltage emission consisting of a  $\text{UO}_2$ -W unidirectionally solidified composite overcoated with  $0.2\text{ }\mu\text{m}$  of  $\text{Al}_2\text{O}_3$  and  $0.2\text{ }\mu\text{m}$  of tungsten. Using electrochemical and chemical techniques, holes will be etched in the tungsten and alumina thin films above each tungsten fiber in the composite. In the fabrication of such a structure, a high resistance  $\text{Al}_2\text{O}_3$  film is necessary to provide the voltage drop necessary for electrochemical etching of the tungsten film. To date, six aluminum discs have been coated with  $0.2\text{ }\mu\text{m}$  of  $\text{Al}_2\text{O}_3$  in an ion beam coater using both 99.9999% purity aluminum and 99.9% purity  $\text{Al}_2\text{O}_3$  as the target materials and  $\text{O}_2$  as the ionized gas. Resistivities of both types of films have been in the range of  $10^3 - 10^5\text{ ohm-cm}$ , and resistivities of the order  $10^8 - 10^{12}\text{ ohm-cm}$  are thought to be required. It is possible that the films are contaminated by iron from the cathode of the ion beam coater. An aluminum cathode is being fabricated in order to test this hypothesis.

### IV. Electron Emission Performance.

The small numbered pin emitter sample prepared in I above (containing approximately 500 pins) was installed in the test diode assembly for emission testing. After a bakeout at  $250^\circ\text{C}$ , pressure stabilized near  $3\text{-}5 \times 10^{-10}$  Torr. Other than this initial system bakeout, no processing was done on the emitter sample.

A large (7 megohm) resistance was inserted in series with the wafer and the anode voltage increased from zero until emission was observed. Emission was slowly increased over about a 48 hour period until a current density of about  $5 \times 10^{-4} \text{ A/cm}^2$  was obtained. Stability of emission current was approximately  $\pm 20\%$ . After operation for about 48 additional hours the stability improved to  $\pm 10\%$ . At this point it was decided to strive for maximum current density, and a current density of  $5 \text{ A/cm}^2$  was achieved with about 5 kV applied to the diode. The water cooling capability of the diode was activated at this time. Operation at the  $5 \text{ A/cm}^2$  current level was continued for about 48 hours after which the current was increased to nearly  $20 \text{ A/cm}^2$ . The current remained relatively constant at this value with short term fluctuations of about  $\pm 10\%$  for 48 hours after which it began to decrease. After three days the current density had decayed 50% and after 5 days to about 10%.

The emission current density observed in these tests was the largest ever obtained from the oxide-metal composites. The fact that these densities were obtained with a small emitter area, and attendant low total anode bombardment is perhaps indicative that anode-initiated destruction mechanisms are of more importance than previously believed.

On 12 February, the sample was removed from the test ring for examination. Preliminary observations with a light microscope revealed no extensive damage to the emitter, but SEM examination is not yet completed.

The rf induction heater previously ordered has arrived and is undergoing installation. This apparatus will be used to thoroughly outgas test diode components in situ at temperatures of  $1000^\circ \text{C}$  and above. Initial tests on conditioned emitters will be performed using an existing glass/metal structure.

MONTHLY TECHNICAL STATUS REPORT

1 February - 1 March, 1974

HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332



# MONTHLY TECHNICAL STATUS REPORT

on

DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the second Monthly Status Report which covers the period from 1 February to 1 March 1974. The report is divided into several sections each covering a different aspect of the work. A Program Schedule and List of Selected Milestones is included in this report.

## I. Preparation of Arrays Having a Small Number of Pins

In the last report, the technique of forming a cone-shaped  $\text{UO}_2$ -W sample using delicate grinding procedures was described. A drawback of this geometry was the presence of fibers along the slope of the sharpened tip which were exposed after etching. In an effort to overcome this disadvantage, polished wafer-shaped samples were mechanically fractured and examined in the binocular microscope. Using this technique it appears feasible to select cylindrical samples consisting of one cell, (approximately 0.025 cm in diameter and several mm long) which should contain about 100 pins. Efforts are under way to explore the etching behavior of these small-numbered pin samples.

## II. Techniques for Selective Etching of $\text{UO}_2$ -W Composites

Copper was conventionally vapor deposited on two discs of  $\text{UO}_2$ -W composites through a 20- and 100-mesh screen producing squares approximately 150 and 830 x 0.5  $\mu\text{m}$  thick. Using silver paste, the samples were attached to 0.5 cm molybdenum pins and etched in chromic acid. No differential etching was observed. The thesis

that differential etching is produced by copper deposits will be reexamined this month on a gross size scale to positively determine the feasibility of this approach. The lack of differential etching may have been due to banded composites, poor electrical contact by the silver paste, formation of a copper oxide surface after deposition, etc.

### III. Low Voltage Field Emission Arrays.

A high purity aluminum cathode was fabricated for the ion beam coater and utilized in the deposition of 2000 Å thick  $\text{Al}_2\text{O}_3$  films on aluminum substrates. Also, 2000 Å thick films of  $\text{SiO}_2$  were produced. The resistivities of all the films tested were still in the  $10^3$  to  $10^5$  ohm-cm range. A rf sputtering unit has been obtained to supplement the ion beam coater as a deposition tool. In the next reporting period reactive sputtering of aluminum will be accomplished. With the much higher deposition rates available in the sputterer, gas contamination should be reduced in the films, hopefully resulting in higher resistivities.

### IV. Electron Emission Performance.

The emitter sample described last month (containing approximately 500 pins and yielding  $20\text{A}/\text{cm}^2$ ) was examined in both the metallograph and the SEM. There was no indication over any of the sample that arcing or electrical breakdown had occurred during emission testing. There appeared to be extensive mechanical damage to the sample. The origin of this damage is uncertain, though it probably originated during handling of the sample after removal from the test diode. The SEM examination also revealed that additional pins were present on the slope of the emitter below the polished point. Such pins could have contributed to the total observed emission current. It is believed

that this contribution was only a small fraction of the total emission because the interelectrode gap was rapidly increasing down the cone-shaped emitter. Efforts to circumvent this problem entail the use of the cylindrical emitter geometry discussed above.

The rf induction heater required for conditioning the emitter has been installed and is in operation. An improved glass/metal experimental tube satisfactory for use with the heater has been constructed. The first satisfactory cylindrical geometry emitter will be tested with this facility and the effect of in situ conditioning will be explored.



TIME-PHASE SCHEDULE FOR THE  
DEVELOPMENT OF A HIGH STABILITY

LONG-LIFE FIELD EMISSION ELECTRON SOURCE WITH A HIGH BRIGHTNESS

NOV	DEC	JAN	FEB	MAR	APR	MAY	JUN	JUL	AUG
Assess Techniques to Obtain 100-Pin Arrays									
Design and Construct Diodes for Emission Tests									
		Experimental Study of Parameters Affecting Field Emission (CBS Phase 1.0)							
		Assessment and (where possible) Experimental Evaluation of Methods to Achieve Single Pin Emitters (CBS Phase 2.0)							
						Evaluation of Field Emission (100 Pin Arrays) and Methods to Obtain Single Pins (CBS Phases 2.1, 2.2)			
						Prototype Emitters for CBS Testing (CBS Phase 2.2)			

## SELECTED MILESTONES

- I. Assessment of Techniques to Obtain 100 Pin Arrays
- II. Construction of Emission Test Diodes
- III. Experimental Study of Parameters Affecting Field Emission
- IV. Assessment and Experimental Evaluation of Methods to Achieve Single Pin Emitters
- V. Evaluation of Field Emission (100 Pin Arrays) and Methods to Obtain Single Pins
- VI. Delivery of Prototype Emitters to CBS

MONTHLY TECHNICAL STATUS REPORT

1 March - 31 March 1974

HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332



# MONTHLY TECHNICAL STATUS REPORT

on

## DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

### SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the third Monthly Status Report, which covers the period from 1 March to 1 April 1974. The report is divided into several sections each covering a different aspect of the work.

#### I. Preparation of Arrays Having a Small Number of Pins

During this period, the majority of effort was concentrated on techniques for polishing and chemically etching very small sliver-shaped samples obtained by fracturing wafer-shaped composites. Samples approximately 0.025 cm in diameter and consisting of one cell were obtained by this method. These samples were glued to a wire-shaped support structure and successfully coated with multiple layers of parafin. After coating, the samples were polished with one-micron diamond paste and selectively etched to expose about 500 fibers each approximately 10  $\mu$ m long. Work is underway to find a way to attach such a small sample to a cathode structure to form an assembly which can be heated to temperatures near 1500 °C. A small jig for mechanically clamping these samples was fabricated; however, it appears that the emitters are too fragile for routine mechanical mounting. The latest approach under consideration is to drill a small hole in a molybdenum pin and support the composite sliver in a platinum braze. It appears that exploratory tests using this method will be performed in the next several weeks.

## II. Low Voltage Field Emission Arrays

Reactive sputtering has been employed in an effort to obtain thin films of approximately 2000 Å thickness having resistivities in the  $10^8 - 10^{12}$  ohm-cm range. A rf sputterer has been employed to deposit several thin films of  $\text{Al}_2\text{O}_3$  on aluminum discs. A target of 99.999% aluminum was fabricated with  $\text{O}_2$  being used as the reactive gas. Since the run was made primarily to ascertain the possible increase in resistivity over previously ion beam sputtered films, no thickness instrumentation was incorporated in the vacuum chamber.

In the initial deposition, the aluminum substrate discs were positioned too close to the target and melting of the substrate resulted from rf coupling. In a later run, the substrate was moved further from the target and the sample remained below the melting point of aluminum. This deposit had a resistance four orders of magnitude higher than any  $\text{Al}_2\text{O}_3$  film previously deposited with the ion beam coater. As a result of this encouraging development, the rf sputterer is being modified to allow greater speed and more precise control of the deposition. A quartz crystal thickness monitor is being installed, and a thermocouple has been added to measure substrate temperature. A new, large diameter, target is being fabricated and an  $\text{O}_2$ -Ar mixture will be employed to increase sputtering speed. As soon as a high resistivity film of known thickness is obtained, etching to produce the desired low voltage emitter geometry will be initiated.

## III. Electron Emission Performance

An emitter sample of about  $0.3 \text{ cm}^2$  area was installed in the glass/metal experimental tube and emission tested. It had been planned to test a composite having a very few pins, but difficulty was encountered in the fabrication and mounting of the physically very small sample. In order to test the basic

experimental facility, the larger sample was installed. Initial bakeout of the entire vacuum system was accomplished at 300 °C. The bakeout oven was then removed and the rf induction heater employed to heat the anode and cathode of the test diode to about 850 °C. This temperature was established as an upper limit because of the copper braze utilized to attach the sample to the molybdenum cathode holder. All other parts of the diode were made from molybdenum and consequently had a much higher temperature limit. Ultimate pressures obtained in the ion pump were  $1-2 \times 10^{-9}$  Torr.

Emission performance of the sample was satisfactory, but not unusual. Performance was comparable to that previously obtained with the same emitter wafer in another apparatus. Emission noise showed no improvement due to the low conditioning temperature.



MONTHLY TECHNICAL STATUS REPORT

1 April - 1 May 1974

HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332

## MONTHLY TECHNICAL STATUS REPORT

on

DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the fourth Monthly Status Report, which covers the period from 1 April to 1 May 1974. The report is divided into several sections each covering a different aspect of the work.

### I. Preparation of Arrays Having a Small Number of Pins

The last monthly report described two alternate methods (mechanical clamping and brazing) that were being considered for the attachment of small sliver-shaped composites to cathode support structures. Initial attempts indicated that the mechanical approach was perhaps too "rough" for the fragile composites and therefore efforts were concentrated on the brazing technique.

Several attempts were made to fasten a composite to a molybdenum support with the use of a platinum braze. Unfortunately, keeping the sample positioned at 1800 °C in a molten pool of platinum was difficult, and in addition, the platinum wet the molybdenum so well that keeping it from crawling out of the small mounting well was impossible. Alternate braze metals were considered, but the high conditioning temperature requirements virtually eliminated most materials with which we have sufficient experience to reasonably expect success. Accordingly, efforts to mechanically secure a sliver-shaped sample were reinitiated.

Numerous attempts to mount a composite sliver in the molybdenum fixture were made before success was obtained. The use of a rhenium foil to support the sample and cushion the clamping action facilitated achieving this geometry.

The sample was successfully coated with parafin, polished, and etched to form an array consisting of approximately 250 pins. This sample was installed in the diode for emission testing.

## II. Techniques for Selective Etching of $\text{UO}_2$ -W Composites

The thesis that differential etching of a  $\text{UO}_2$ -W composite may be produced by a copper deposit was reexamined this month on a gross scale compared with the size needed for a high brightness field emitter. A disc of  $\text{UO}_2$ -W composite approximately 1.3 mm thick and 3 mm in diameter was polished to a 1 micron diamond finish on both sides and copper brazed to a 1.5 mm diameter molybdenum cylinder. After brazing, the sample was immersed for a few seconds in dilute nitric acid to remove excess copper surrounding the moly pin. It was noticed that the nitric acid etched the  $\text{UO}_2$  on the front surface except for the area directly above the copper deposit. This area retained the original polish. The sample was etched in the normal 3:2 chromic acid etch for two hours. Etching was, as expected, confined to only the areas not backed by copper. The walls of the unetched area above the copper sloped to the center of the pin because the etch was slowly dissolving the copper braze and decreasing the area protected by the copper. The central portion of the sample above the center of the moly pin had vertical walls and the surface was unetched.

This demonstrates that composite samples could be copper brazed to molybdenum pins of small cross sectional area and preferentially etched to produce small diameter  $\text{UO}_2$ -W composites. The use of copper is a decided disadvantage of this technique since it appears necessary to heat emitters to 1500-1800 °C which would vaporize the copper. For the present, preferential etching will not be further pursued unless it is shown that high temperature processing is not necessary for emission stability.

### III. Low Voltage Field Emission Arrays

The past month has been devoted to modifying the rf sputterer to achieve control over thin  $\text{Al}_2\text{O}_3$  film deposition. Thermocouples and a thickness monitor have been installed in the sputterer. Development of high resistance films will continue this month.

### IV. Electron Emission Performance

The sample containing approximately 250 pins was installed in the test diode and baked at  $200^\circ\text{C}$  for about 36 hours. After cooling the pressure was about  $1.5 \times 10^{-8}$  Torr. At this point electrical testing was started. Initial current was very low until the anode potential reached 12 kV, at which value the current increased greatly. After this "activation" 3.2 nA/pin could be obtained with about 3.8 kV applied. Current remained at this value with no apparent decrease for about 7 days at which time processing was initiated. During the pre-processing period, the observed noise was about 10% peak-to-peak of the average current.

Processing was accomplished by heating the emitter and anode structures to approximately  $1000^\circ\text{C}$  for a period of about 16 hours. The anode voltage was reapplied immediately after shutoff of the rf heater. Pressure at this time was about  $3 \times 10^{-8}$  Torr. The current observed when anode voltage was reapplied had a much smaller noise component of less than 1% peak-to-peak. The post-processing voltage required to produce emission currents comparable to pre-processing was up by about 100%. This increase is attributed to rounding of the pin tips during processing. I-V curves taken after processing have been the most reproducible ever obtained. At the present time (approximately 300 hours of operation) emission current has been increased to 26 nA/pin with no significant increase in noise.

It is planned to continue noise and conditioning experiments during the coming month.

**MONTHLY TECHNICAL STATUS REPORT.**

**1 May - 1 June 1974**

**HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS**

**Subcontract for AF Prime Contract**

**F08606-73-C-0028**

**CBS Laboratories No. 52501**

**School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332**



# MONTHLY TECHNICAL STATUS REPORT

on

## DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

### SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the fifth Monthly Status Report which covers the period from 1 May to 1 June 1974. The report is divided into several sections each covering a different aspect of the work. A Program Schedule and List of Selected Milestones is included in this report.

#### I. Preparation of Arrays Having a Small Number of Pins

The last monthly report described the successful fabrication and mechanical mounting of a small "sliver-shaped" composite containing approximately 250 pins in a cathode structure. The emission performance of this sample, designated CBS-2, was described last month and in Section IV of this report. It is most noteworthy that a significant decrease in emission noise was obtained by heating (processing) this sample in vacuum at about 1000°C, a much lower temperature than anticipated.

Since the emission testing of CBS Sample No. 2, numerous efforts have been made to fabricate additional composite emitters from "sliver-shaped" samples. This work has included a repeat of the mechanical clamping technique used with CBS-2, as well as brazing composite slivers in platinum foil and platinum foil lined with copper. Unfortunately, neither of these approaches can be accomplished easily and, in either case, the minimum number of pins obtainable is several hundred.

An alternative approach is under consideration to utilize a micro-manipulator in the scanning electron microscope (SEM) to mechanically shave all but a few pins flush with this surface of the composite. This

instrument has a translation resolution of several microns. This approach appears promising since samples have been successfully shaved using a razor blade in conjunction with a light microscope to form arrays consisting of several hundred pins. Such a geometry would then have to be overcoated, except for the standing pin(s), with another material such as alumina or tungsten to eliminate emission from the fibers flush with the surface. Mr. Capp Spindt of SRI has been contacted about the prospects of using his microfabrication techniques to obtain coating except over the single or few pin region. A letter describing this interest and need will be forwarded to SRI with copies to CBS next week. Attached as letter appendix to this report is a request for a contract supplement of \$5000.00. These funds are for the purchase of the micro-manipulator for the SEM and several hundred hours of operator time.

Since it has been experimentally demonstrated that the noise characteristics of the oxide-metal composite emitters are approximately the same for a 200 or  $2 \times 10^6$  pin sample, a new emitter is being fabricated (designated CBS-4) which has excellent growth characteristics and is approximately 0.5 cm in diameter. This sample will be used to appraise the effectiveness of the in situ heat treatment on the noise characteristics of the composite, but due to the copper braze, processing will be limited to approximately 950°C. However, since this temperature was very effective in improving the noise behavior of the CBS-2 sample studies of the time-noise characteristics as well as the emission behavior after exposure to air are planned.

## II. Techniques for Selective Etching of $UO_2$ -W Composites

Because of the progress last month in emission performance after processing at 1000°C (reported in Section IV of the April Monthly), it may be possible to use copper as a braze for the emitter. Therefore, an attempt was made to make a sample 40 - 80  $\mu$ m in diameter by selective etching.

One end of a 6 mm molybdenum rod was machined to a 0.8 mm diameter and the tip was sanded to a 120 micron diameter. A ceramic jig was fabricated to hold the 120 micron tip on a  $\text{UO}_2$ -W composite wafer with a small amount of copper sandwiched between the moly pin and the composite. The jig containing the moly pin and composite was heated to  $1100^\circ\text{C}$  for 10 minutes and slowly cooled. Brazing was not accomplished due to vaporization of the copper. Once again copper brazing has been discontinued as an approach for producing small diameter composite samples.

### III. Low Voltage Field Emission Arrays

During the past month, a vacuum leak developed in the rf sputterer used for deposition of  $\text{Al}_2\text{O}_3$  films. The leak was finally traced to a water cooling line and the cooling line was replaced. The system now can be evacuated to  $2 \times 10^{-6}$  Torr with the diffusion pump only and with liquid nitrogen the system should reach the  $10^{-7}$  Torr range. Development of high resistance films will continue this month.

### IV. Electron Emission Performance

The last monthly report described the initial emission performance of the composite sample, CBS-2, containing 250 pins. Testing of this sample was continued this reporting period and Figure 1 shows a current versus time graph for the conditions imposed on this sample. As noted earlier the noise characteristics of the sample decreased drastically after the heating at  $1000^\circ\text{C}$  for approximately 16 hours. Recorder charts showing peak-to-peak current variations and a V-I curve typical of this sample were forwarded to CBS earlier this month.

After processing an attempt was made to increase the current per pin up to approximately  $1\mu\text{A}$ . This information is shown in Figure 1 as the performance accrued between days 18 and 22 of testing. Unfortunately, when

the current reached approximately 400nA per pin, the sample was apparently mechanically lifted from the cathode structure by the electrostatic force. Consequently, the "sliver-shaped" emitter was not available for post emission analysis and, in fact, we were unable to find it during the disassembly of the vacuum chamber.

The noise characteristics during days 19 through 22 of testing were still significantly better than that noted during the preprocessing period. There appeared, however, to be a gradual increase in the noise characteristics during this time period.

Three significant results obtained from the emission testing of CBS Sample 2 are:

1. The noise characteristics of a 200 or a  $2 \times 10^6$  pin sample are essentially the same, at least in the preprocessed state,
2. Heating in vacuum greatly reduces the noise characteristics of the oxide-metal composite emitters and processing at temperatures as low as  $1000^{\circ}\text{C}$  appear feasible, and
3. The recontamination of the pin tips, even in a vacuum of  $10^{-8}$  atmospheres, is much slower than originally expected and "low noise" emission has been observed for periods up to 250 hours at currents of several hundred nA per pin.

During this report period, a test of the effects of an electropositive adsorbate (barium) on the emission performance was undertaken. The sample involved, designated CBS-3, has approximately 1 million W fibers of  $7\mu\text{m}$  height distributed with a density of  $\sim 8$  million fibers  $\text{cm}^{-2}$ .

The barium vapor source and test sample are enclosed in an all-metal vacuum system that has no provision for rf heating. No means of heating



the fibers is available, so that it should be assumed that most of the fibers are covered with several monolayers of contaminants. If all of the fibers were so contaminated they would not experience a work function decrease by barium absorption and, therefore, no (significant) change in the emission behavior of the sample should be expected with the admission of barium vapor to the vacuum chamber. However, the possibility exists that some fraction of the fibers emit so strongly that, through resistive heating, their temperature become elevated to a level sufficient to reduce the contaminant coverage to a degree that allows the fibers to "see" the barium. Hence, the possibility that the addition of barium vapor to the vacuum chamber result in an increase of emission does exist, becoming more likely as the sample current is increased.

Approximately 165 hours after the initiation of sample testing, the first attempt to coat the sample with barium was made. Prior to this time, seven sets of V-I data were obtained and plotted in Fowler-Nordheim (FN) fashion for comparison with a V-I data set to be taken immediately after the attempt. No increase of emission was observed when the barium vapor was let into the system and no change of emission characteristics observed on the FN plots.

The total array currents prior to and during the first barium test, being of the order of  $100\mu\text{A}$ , were probably not large enough for any of the fibers to clean themselves enough to see the barium. So, for about 170 hours after the first barium test, the array current was increased to  $1\text{mA}$ , a V-I data set was obtained (and put on an FN plot), and a second barium test was conducted. Again, no increase in emission was observed during the test and the FN plot of a V-I data set taken immediately after the test was essentially the same as the one made before the test. Similar results were obtained with the third barium test (taken some 160 hours after the second one) in which the average current was  $10\text{nA/pin}$ . The diode system utilized



for this sample is not capable of properly dissipating much more power, so we do not expect to perform any more barium tests on this sample.

At the time of this writing, the sample has been operated continuously for slightly more than 750 hours. For the past 350 of these hours the sample has been operating at 10nA/pin with remarkable short-term (5-7 hours) stability. An occasional drift of the current to 11 or, less often, 12 nA/pin is observed, but these drifts are very slow -- no erratic fluctuations have been observed.

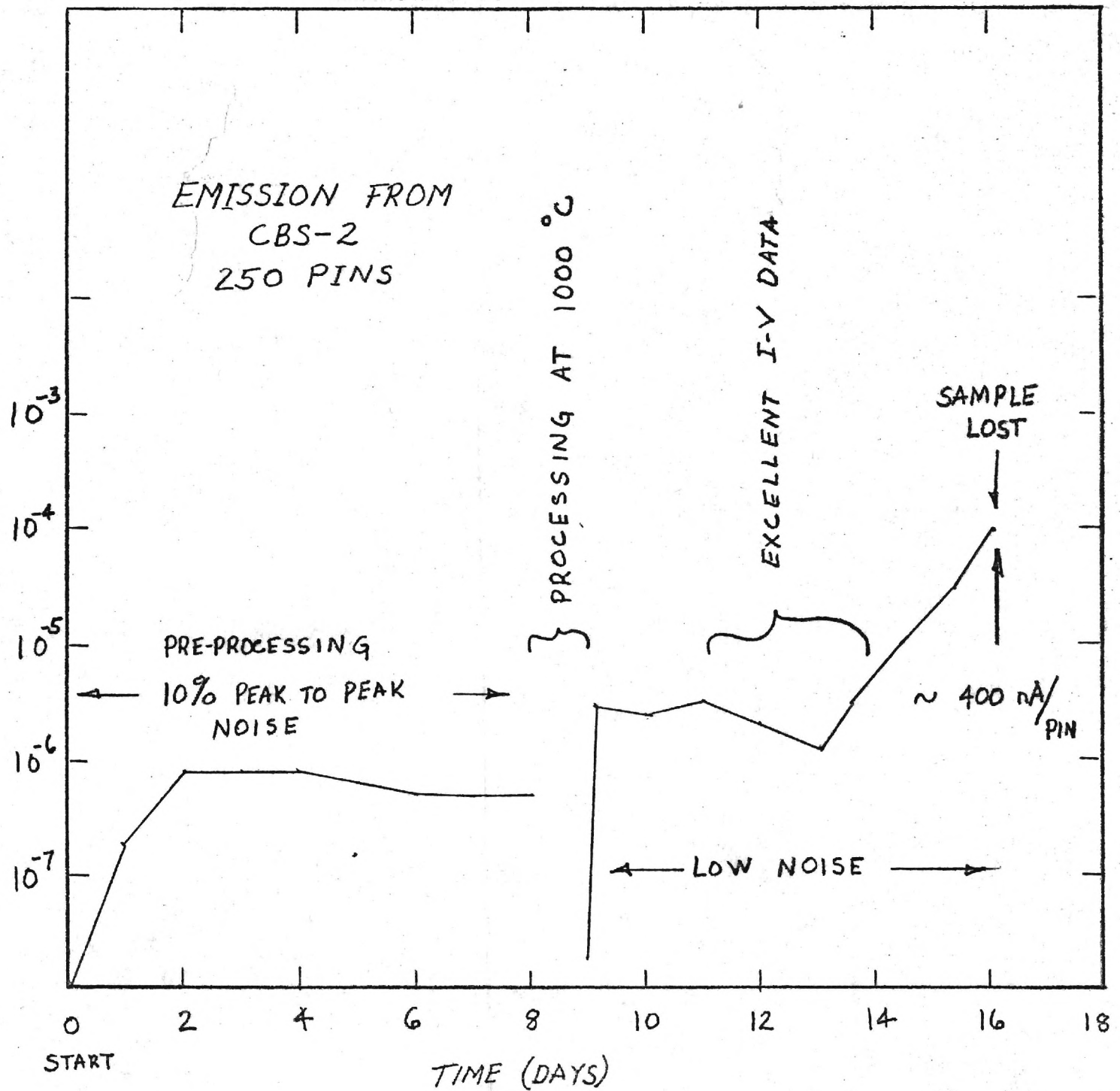


Figure 1. Emission from CBS-2 as a Function of Time.

MONTHLY TECHNICAL STATUS REPORT

1 June - 1 July 1974

HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332

# MONTHLY TECHNICAL STATUS REPORT

on

DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the ~~sixth~~ <sup>seventh</sup> Monthly Status Report which covers the period from 1 June to 1 July 1974. The report is divided into several sections each covering a different aspect of the work.

## I. Preparation of Arrays having a Small Number of Pins

As noted last month, there was no significant difference between the field emission noise characteristics of oxide-metal composite samples containing 200 or  $2 \times 10^6$  pins. Consequently, the effect of heating on emission behavior is being conducted on samples with large number of pins because of their ease of fabrication. The effort to obtain a small numbered array is temporarily interrupted until the micro-manipulator for use in the scanning electron microscope (SEM), which has been ordered, is received. This instrument will be used to "shave-off" all but a few pins flush with the surface with the composite. (The justifications for using this approach was described in some detail last month.)

## II. Low Voltage Field Emission Arrays

Since the last monthly report considerably improved control of the rf sputterer has been achieved. Aluminum oxide films have been deposited using an aluminum target and an argon-10% oxygen gas mixture. Several deposits have resulted in resistivities for the aluminum oxide films of  $10^7$  to  $10^8$  ohm-cm.

Literature review has shown that alumina films of equal quality may be deposited by using alumina as the target and pure argon as the ionized gas and that higher deposition rates may be achieved. Consequently, an alumina target of 10 cm diameter and 0.5 cm thick has been installed in the sputterer with improved coupling of the rf field to the gas.

Thickness monitoring of the deposits using a vibrating quartz crystal is still a problem. Either the rf field is creating spurious signals or the quartz crystal is heating excessively. Both additional electrical shielding and increasing the distance between the crystal and the target will be attempted. At the present time the weight of a glass slide is being measured before and after each run to determine thickness. This will be continued until satisfactory results are obtained with the piezoelectric thickness monitor.

During this month, deposition of tungsten films on the alumina will be initiated along with etching procedures to produce a low voltage field emission array.

### III. Electron Emission Performance

This section describes the electron field emission behavior of emitter sample designated CBS-4, which was designed to further appraise the effectiveness of in situ heating on the noise characteristics of the oxide-metal composites. Sample No. 4 was a  $\text{UO}_2$ -W composite copper brazed onto a 3/16th inch diameter molybdenum rod. The sample contained approximately 1.4 million pins in the shape of right circular cylinders  $8\mu\text{m}$  in length, with excellent pin uniformity.

One interesting observation was made during the preparation of this sample. After chemical etching, the sample was untrasonically cleaned in benzene and methanol for several hours. The prolonged cleaning resulted in



the loss of fibers in large areas. Apparently ultrasonic cleaning caused enough pin vibrations to "work-harden" the delicate pins which broke off at the matrix surface.

After re-etching and non-ultrasonic cleaning the sample was installed in the test diode with a spacing of approximately 0.125 inches. The diode was baked at 150-200°C using electrical tapes and ion pumped resulting in a system background pressure of  $2 \times 10^{-8}$  Torr. Emission was readily accomplished by applying an anode voltage of 7.5 kV with a resulting gross current of approximately  $10^{-6}$  A. The current was intentionally kept low prior to heating (processing) in order to evaluate the effectiveness of processing since higher current levels are suspected to have some self-cleaning action. Typical noise was about plus or minus 10% of the average current.

After three days of operation at this current level the anode voltage was turned off and the sample was heated to 850°C. During heating there was significant system pressure rise and the sample was held at this temperature overnight; the following morning the base pressure had returned to the pre-heating value  $2 \times 10^{-8}$  Torr. When the sample was reactivated it was most noteworthy that the gross current of  $10^{-6}$  A was easily obtained again, but at an anode potential of only 4.3 kV. The noise was significantly lower than that noted before processing. The noise characteristics while greatly reduced, as compared to the preprocessed condition, were not as low as that observed during the emission testing of the sample, containing approximately 200 pins, (Sample CBS-2). After operating under this condition for about 6 hours the anode voltage was turned off and further processing designed to reach 1000°C was undertaken. The sample was heated to 1000°C with only a small rise in the system pressure and left at this temperature. Unfortunately, the temperature of the cathode slowly increased to above 1000°C.

The processing continued for two hours at which time the pressure had dropped into the low  $10^{-8}$  Torr range. When the anode voltage was reapplied it required 10 kV to reach a gross current of  $5 \times 10^{-7}$  A, and the current stability was very poor. Operating the sample under these conditions overnight showed a modest improvement in noise but no increase in the emission current.

Because of the deterioration in the emission performance of this sample it was removed for examination. Both optical and SEM observation indicated no deterioration of the pins, in fact, the processing at  $>1000^{\circ}\text{C}$  may have rounded the edges of many of the pins. A small area of the copper braze between the composite and Mo support pin was melted and had welded portions of the cathode assembly together making disassembly difficult. Copper was also found on the anode but no copper was evident on the tungsten pins. At present there is no obvious explanation for the gross deterioration in emission performance of this sample (CBS-4).

In addition to emission testing this report period significant improvements on the pumping and diode assembly were made which should reduce turn-around-time for testing future samples.

At the time of the writing of the last monthly report, sample CBS-3 had acquired a continuous operating time of approximately 750 hours. Since that time, it has required an additional continuous operating time of 825 hours bringing the present total to 1575 hours. All but the first 400 hours of this sample's operation have been at a current per pin level in the neighborhood of 10nA ( $\sim 10\text{mA}$  total emission). As noted last month, the sample has exhibited remarkable stability in current with the only noticeable noise being a long-term drift of 1 to 2nA per pin. The fluctuations in the current over any 15-20 hour period have typically been less than  $\pm 1\%$ .

MONTHLY TECHNICAL STATUS REPORT

1 July - 1 August 1974

HIGH STABILITY LONG LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332

## MONTHLY TECHNICAL STATUS REPORT

on

DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the eighth Monthly Status Report which covers the period from 1 July to 1 August 1974. The report is divided into several sections each covering a different aspect of the work. A Statement of Accumulated and Monthly Expenditures is included in this report.

### I. Preparation of Arrays Having a Small Number of Pins

Authorization was obtained from CBS to purchase a micro-manipulator for the SEM (to be used for the fabrication of the small number pin arrays). Unfortunately, there was a four week delay in ordering this equipment; it was initially ordered as a supply item, but the contract administrators at Georgia Tech required CBS authorization and a capital equipment expenditure. The manufacturer of the micro-manipulator has been informed that an order will be forthcoming shortly, in hopes that he will initiate the fabrication of this equipment and minimize the delivery delay.

A letter has been sent to SRI inquiring as to the usefulness of their "micro-deposition" techniques with the geometries expected using the micro-manipulator in the SEM. Copies of this letter were forwarded to CBS.

### II. High Temperature Braze Development

A continuing small effort has been underway to investigate improved brazing materials and techniques for use with the oxide-metal composites.

A most noteworthy development obtained this reporting period was the satisfactory use of platinum to braze the composite materials. Improvements in the cleaning techniques, and the application of the braze, and uniformity of temperature control apparently were responsible for a satisfactory platinum bond between  $\text{UO}_2$ -W composites and Mo pins. Platinum brazed samples should enable processing to temperatures approaching  $1800^\circ\text{K}$  prior to emission. It has been reported that temperatures of this magnitude are needed to desorb tightly held oxygen atoms from the tungsten surface. It is anticipated that the next emission test sample, designation CBS-6, will have a platinum braze. The comparison of the emission characteristics as a function of processing temperature will then be possible. One area of uncertainty which will have to be investigated, is the effect of the Pt braze on the etching characteristics of the composites.

### III. Low Voltage Field Emission Arrays

A reproducible technique for measuring the resistance of  $2000 \text{ \AA}$  dielectric films was developed in the past month. Conventional vacuum evaporation was used to deposit 1000 to  $2000 \text{ \AA}$  of aluminum on  $2.5 \times 7.5 \text{ cm}$  glass slides. Alumina was sputtered over the aluminum film to the desired thickness ( $1000$ - $2500 \text{ \AA}$ ). Edges of the slide were masked during alumina deposition to expose the aluminum bottom electrode. Aluminum squares,  $8\text{mm}$  on a side, were evaporated over the alumina film to serve as the top electrode. Small aluminum wires were silver pasted to the top and bottom electrodes for electrical contact.

Two runs have been made using this technique. Efficiency of the first was low (one good film out of six), but on the second trial, nine out of ten of the  $8\text{mm}$  squares had resistivities of  $> 10^{14} \text{ ohm cm}$ . The increased



efficiency was probably due to better cleaning of the glass slide prior to deposition and improved handling techniques to promote cleanliness at every step of the process. The glass slides were scrubbed in a detergent, rinsed in distilled water, ultrasonically cleaned in concentrated hydrochloric acid, followed by a rinse in distilled water and a final rinse in methanol.

Apparatus consisting of electrolytic cell, a pulse generator, a voltage source, and an oscilloscope has been set up to etch holes in 2000 Å tungsten films above the fibers in the oxide metal composites. A brief review of the sandwich structure and desired results is appropriate at this point. Referring to Figure 1, the structure will consist of a 2000 Å alumina film over the  $UO_2$ -W composite with 2000 Å of tungsten over the alumina film. By using the sandwich structure as the positive electrode in a KOH electrolysis cell, the tungsten may be oxidized and subsequently dissolved in the KOH solution. (Tungsten is insoluble in aqueous KOH). Anodization of the tungsten will initiate directly above the tungsten fibers of the composite, since this will be the path of lowest electrical resistance. If the anodized tungsten oxide is not dissolved at a faster rate than it is formed, the oxide layer will spread over the surface and the holes will be much larger than the fibers. This hopefully will be prevented by pulsing the voltage to the cell long enough to form localized oxide layers above the fibers and removing the voltage long enough to dissolve the anodized tungsten. Using the oscilloscope, initiation of the anodization hopefully will be detected by an increase in voltage across the cell and a decrease in current. This will allow determination of the proper length of the voltage pulse.

Two composite samples have been prepared with a  $\approx 2000$  Å  $Al_2O_3$  film overcoated with 4000 Å of tungsten. Studies of etching to produce the holes will be initiated this week.

#### IV. Electron Emission Performance

This section briefly describes the chronological emission performance of CBS Sample No. 5. This emitter was fabricated by simply repolishing the CBS-4 sample. It had excellent growth characteristics, contained approximately  $1.8 \times 10^6$  pins  $8\mu\text{m}$  long and was copper brazed to a 3/16" diameter Mo rod. The interelectrode spacing was 0.1 inch, approximately the same as that used in experiment CBS-4.

During the initial application of anode voltage to the sample, 5kv was required to initiate detectable emission. Emission immediately increased and at an applied potential of 2.5kv a gross emission current of  $5 \times 10^{-7}\text{A}$  was obtained. The noise characteristics during this early testing were surprisingly low with peak-to-peak current variations of  $\pm 10\%$  observed. The sample remained at this applied voltage for 24 hours during which time emission decayed to approximately  $2 \times 10^{-7}\text{A}$  and the noise characteristics decreased to  $\pm 5\%$ . The applied voltage was increased to 4.5kv with a resultant current of  $5 \times 10^{-6}\text{A}$ . The noise characteristics were still low for a sample which had undergone such a short period of emission testing. After an additional 12 hours at this applied voltage, the current had decreased to  $\approx 1 \times 10^{-6}\text{A}$ . The short term noise fluctuations had decreased during this period to almost zero (as monitored on a strip chart recorder); however, at three to five minute intervals, the sample would make current excursions of  $\pm 100$  to 200%. Additional noise traces were run in the  $10^{-7}$  to  $10^{-8}\text{A}$  current range, and for some reason contradictory to most of the previous emission experiments, the noise was less, and appeared more stable at the lower current levels.

The vacuum chamber was next opened to the atmosphere for several hours and then the ion pumping was restarted. This procedure was designed to see

the effect on the emitter noise characteristics of exposing the W pins to air. When field emission was reinitiated after pumping for 48 hours, the sample displayed current excursions of  $\pm 500$  to 1000%. Even after an additional twelve hours of operation at this level, variations were still  $\pm 100$  to 200%.

V-I data just prior to the initial processing (heating) of Sample CBS-5 showed 5.5kv required to obtain  $4 \times 10^{-6}$  A with current variations of  $\pm 50\%$ . Processing of the sample over-night at temperature below  $700^{\circ}\text{C}$  lowered the voltage to 3.8kv required to achieve  $\sim 5 \times 10^{-6}$  A. The decreased anode potential required to obtain equivalent pre-processing emission current is similar to the behavior observed during the testing of CBS-4. The noise characteristics of CBS-5 after processing were less than in the preprocessed condition, but was still  $\pm 10$  to 20%. During the twenty hour period after the  $700^{\circ}\text{C}$  processing, a slow increase in applied anode potential was needed to maintain the current at  $\cong 5 \times 10^{-6}$  A. After twenty hours, 5.3kv yielded a  $5 \times 10^{-6}$  A with noise characteristics of  $\pm 10$  to 20%. This decay in emission performance was probably the result of readsorption of surface contamination on the tungsten pins.

The second processing was then initiated at  $850^{\circ}\text{C}$  and heating continued for 24 hours. It was observed that a substantially higher voltage was required to produce emission. After cooling, when the anode spacing had returned to its initial preprocessing length, 9.5kv was required to obtain an emission of  $5 \times 10^{-6}$  A. The post-processing noise was drastically reduced and exhibited at most  $\pm 1\%$  current fluctuations. After seven hours of operation, the noise had increased to  $\pm 6\%$ .

Since some uncertainties were present because of instrumentation and power supply problems, processing was resumed and held an additional 15 hours at  $850^{\circ}\text{C}$ . Post-processing emission characteristics were similar to that

obtained previously. The noise remained at 5 to 8%, however, there was noticeable increase in the kv required to achieve a current of  $5 \times 10^{-6}$  A. This required a change in power supplies and the application of  $\approx 13$ kv to obtain this current.

It was noted that after processing was completed, the noise characteristics were generally low as long as significantly higher voltages were not required to obtain the same current levels. Thus, an emission current of  $10^{-8}$  to  $10^{-7}$  A was relatively noise free since this current level could be obtained below 10kv, whereas, the noise (even post-processing) at  $5 \times 10^{-6}$  A was significant because 13kv was required to obtain this current density. Prolonged heating of the composites at temperatures above  $800^{\circ}\text{C}$  apparently caused rounding of pin edges which greatly increased the anode potential required to obtain emission. Obviously, geometry and surface changes are occurring simultaneously during this low temperature processing treatment and definite results are difficult to achieve. Several additional processing cycles were completed on the CBS-5 sample and the general emission behavior followed this same pattern.

After these processing treatments, the sample was subjected to increased anode voltage in order to bring the current per pin up in the nA range. The anode voltage was increased to 16kv with current variations of  $\pm 200$  to 300% common. After an additional 130 hours, during which time the anode potential was continually reduced, the sample was operating at  $4 \times 10^{-4}$  A or  $\approx 1$ nA/pin at an applied voltage of 12.3kv. The noise characteristics had reduced (apparently by some type of self-cleaning mechanism) and the short term fluctuations were almost nonexistent, but at two or three minute intervals, current fluctuations of  $\pm 5$  to 10% were present producing the typical box appearance on the strip chart recorder. Emission testing of this sample is



continuing.

The long term emission testing of CBS Sample No. 3 has continued through the month of July. The sample now has a total of about 1925 operating hours at a current level of 10 nA/pin. There has been no sign of current decay during this period and the noise has remained less than  $\pm 1\%$ . The sample has been subjected to occasional cycling on and off for periods of a few seconds to several hours without any apparent change in emission characteristics.

#### V. Financial Report

Expenditures through June 1974 are approximately: \$ 7,494.65

Expenditure for July will be approximately: \$ 8,000.00

Total \$ 15,494.65



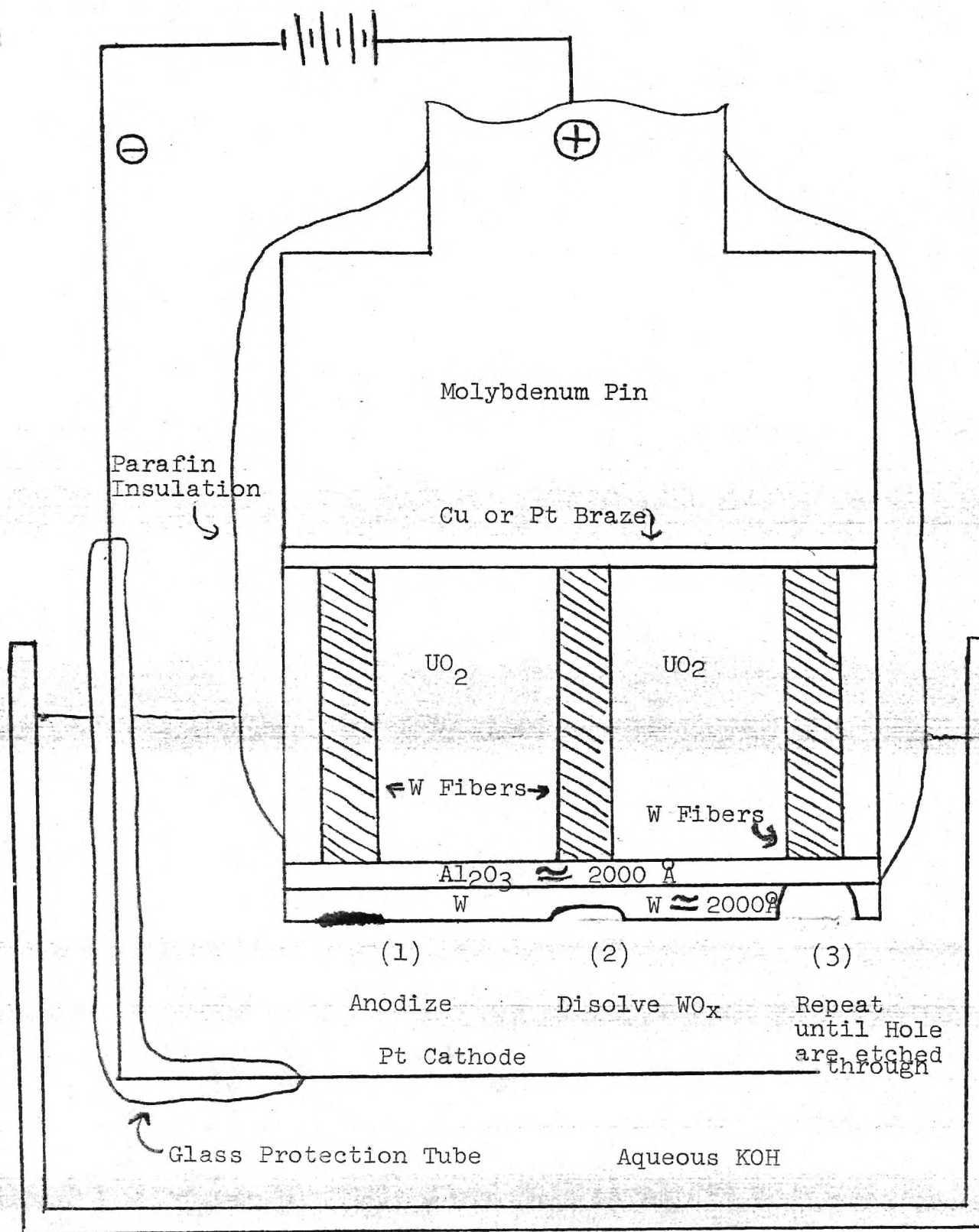


Figure 1. Anodization Cell for Oxidizing Tungsten and Dissolving Tungsten Oxide

MONTHLY TECHNICAL STATUS REPORT

1 August - 1 September 1974

HIGH STABILITY LONG LIFE FIELD EMISSION ELECTRON

SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract

F08606-73-C-0028

CBS Laboratories No. 52501

School of Electrical Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332

# MONTHLY TECHNICAL STATUS REPORT

on

## DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the ninth Monthly Status Report, which covers the period from 1 August to 1 September 1974. The report is divided into several sections each covering a different aspect of the work.

### I. Preparation of Arrays Having a Small Number of Pins

Work in this area is temporarily suspended pending the arrival of the "Micro-Manipulator" for use in the SEM. Phone conversation with the supplier indicates it will not be until mid November before this item is manufactured, and consequently, only a short trial period will be available prior to the anticipated completion of the contract on 31 December 1974.

No answer has been received from the SRI indicating their interest in using micro-deposition techniques with the oxide-metal composites.

The fabrication of CBS Sample No. 6 using a platinum braze has been completed and the sample installed in the test diode. (Some details of diode modification and improvements are given in the Electron Emission Performance section of this report.) This sample has  $50 \times 10^3$  exposed pins on a cylindrically shaped sample approximately 4mm tall and 1mm in diameter. Unfortunately some etching problems still exist with platinum brazed samples and this sample has a uniform pin array over approximately 50% of the surface; the remainder of the area is unetched.

## II. Low Voltage Field Emission Arrays

Etching studies to produce holes in a tungsten film above each fiber in a  $\text{UO}_2$ -W composite were initiated this month. Three samples were subjected to a positive four to five volts in a 15-normal KOH solution. Voltage was supplied by a constant voltage source so that current could be used as the indicator of oxidation of the W film. Voltage pulses of 500 and 120 msec with a 2500 msec cycle were used on the three samples. At the start of the etch, current through the sample was approximately 500 mA and the current decreased with each cycle, reaching a level of 10 mA after approximately 10 cycles. The first sample was etched for two hours total time and the tungsten film was completely destroyed. The second sample ran for one hour with the same results. For the third sample, the voltage pulse was decreased from 500 to 120 msec and run for 35 minutes. Again the film was 90% removed, but in one area the film remained and it did contain holes or depressions corresponding to the tungsten fiber geometry in the composite. This was encouraging. It became obvious that a large number of samples are needed for the etching studies and a student in Chemical Engineering has been employed to produce these composite samples.

The pre-etch condition of the tungsten films was less than desirable and probably contributed to the destruction of the films during etching. There appears to be no problem with the 2000 Å alumina films applied to the surface of the  $\text{UO}_2$ -W composite, but when the alumina is overcoated with tungsten, small areas are seen where the W and  $\text{Al}_2\text{O}_3$  film has been removed. It is thought that compressive failure is resulting as the samples cool after tungsten sputtering due to thermal expansion mismatch. During sputtering the substrate is heated to between 150 and 200°C.

Compressive stresses in the tungsten were estimated from:

$\sigma = E(a_{\text{film}} - a_{\text{composite}})\Delta T$ , where  $\sigma$  is the resulting stress in the tungsten film,  $E$  the modulus of elasticity of tungsten,  $a_{\text{film}}$  and  $a_{\text{composite}}$  are the thermal expansions of tungsten and the  $\text{UO}_2$ -W composite respectively, and  $\Delta T$  is the temperature differential the sample experiences during cooling. Using typical values of

$$E = 50 \times 10^6 \text{ psi}$$

$$a_{\text{film}} = 5 \times 10^{-6} / ^\circ\text{C}$$

$$a_{\text{composite}} = 10 \times 10^{-6} / ^\circ\text{C}$$

$$\text{and } \Delta T = 200^\circ - 20^\circ = 180^\circ\text{C},$$

a resulting 45,000 psi compressive stress is calculated. Therefore, with a stress level of this magnitude the defects seen are certainly due to expansion mismatch.

The stresses may be eliminated by reducing the expansion differential or by reducing the temperature differential. The latter has been chosen. Tungsten films will be deposited with an ion beam coater which, although slower than sputtering, does eliminate sample heating during deposition.

### III. Electron Emission Performance

Last month's report described in detail the emission performance of CBS Sample No. 5. During this report period the sample operated an additional 100 hours and was running overnight with a recorder monitoring the noise characteristics when at 11:00 p.m. a fire destroyed the power supply. Enough heat was generated to melt the glass envelopes on the vacuum tubes and ignite the adjacent recorder chart paper. As a result of the dense smoke from the plastic components in the power supply, the security



personnel were forced to spray the entire laboratory with dry chemicals. As a result of the needed clean up several weeks of experimental time was lost.

During this period a longer glass diode was installed under to dissapate the increased heat generated with the higher processing temperatures. A factory new ion pump was installed and a new anode structure was fabricated with the arc-fused moly rod recently supplied by CBS.

Post-emission SEM examination the CBS-5 Sample showed no obvious pin damage and some apparent rounding of the cylindrically-shape pins.

The platinum brazed sample, CBS-6, has been installed in the diode and bakeout is in progress. It is anticipated that this sample will be initially subjected to a high temperature processing approaching 1400°C, and then the anode voltage necessary to draw 5-10nA per pin will be immediately applied. It is anticipated this treatment will minimize or eliminate the current excursions associated with the low temperature processing (1000°).

The long term emission testing of CBS Sample No. 3 continued through the month of July and the sample has now logged a total of about 2650 hours. The noise remains less than 1% and the sample has been continually subjected to almost daily cycling on and off for periods of a few seconds without any apparent change in emission characteristics.

#### IV. Financial Report

Expenditures through September 1974 are approximately:	\$27,223.35
Estimated expenditures for October will be:	\$12,000.00
Total :	\$39,223.35

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MONTHLY TECHNICAL STATUS REPORT

1 September - 1 December 1974

HIGH STABILITY LONG LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract  
F08606-73-C-0028

CBS Laboratories No. 52501

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MONTHLY TECHNICAL STATUS REPORT  
on  
DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION  
ELECTRON SOURCE WITH A HIGH BRIGHTNESS  
CBS LABORATORIES NO. 52501

This report is the combined tenth, eleventh and twelfth Monthly Status Report, which covers the period from 1 September to 1 December 1974. The report is divided into several sections covering different aspects of the research program.

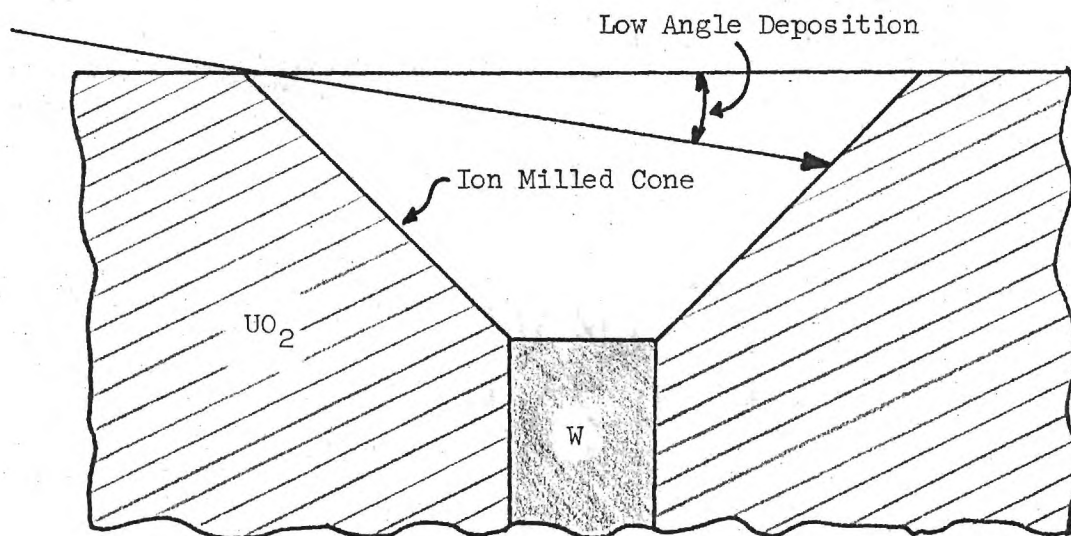
I. Preparation of Arrays Having a Small Number of Pins.

The "Micro-Manipulator" was received in the middle of November and modifications are under way to further improve the sensitivity of lateral movement. It is anticipated preliminary trials at shaving the pins on composite samples will be accomplished and reported next month.

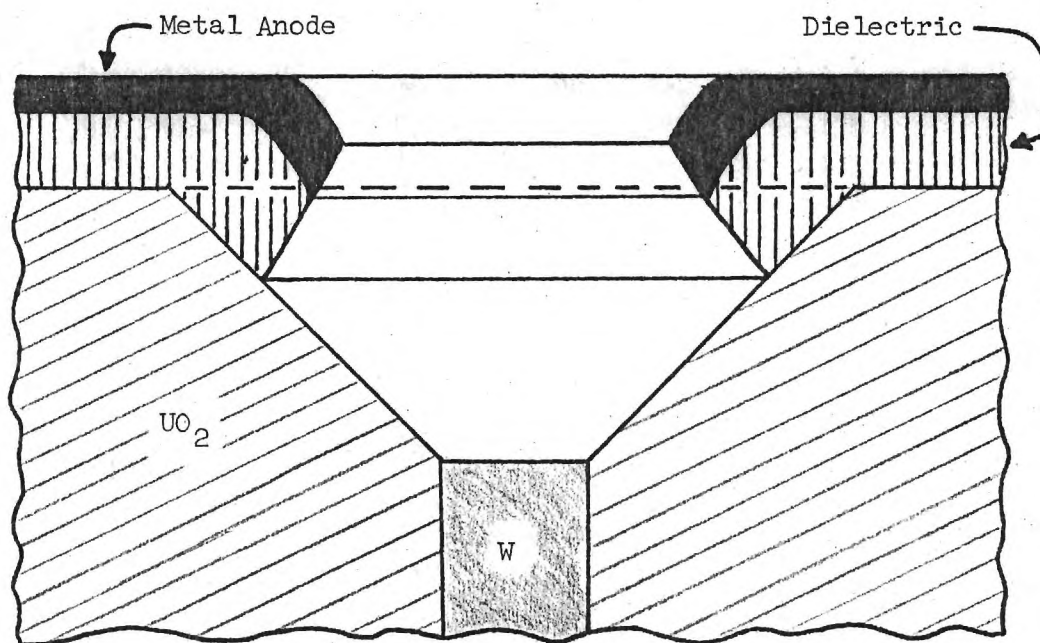
## II. Low Voltage Field Emitters

A second method for fabricating low voltage emitter sandwich structures has been developed in the last three months. It was discovered that argon ion milling a polished  $\text{UO}_2$ -W composite would recess the W fiber into the  $\text{UO}_2$  matrix with the fiber centered in an inverted truncated cone, Figure 1. To produce a sandwich structure, a dielectric and a metal may be vapor deposited sequentially at a sharp angle to the ion milled surface while the composite is rotating. This should evenly coat the surface of the  $\text{UO}_2$  and the outer part of the ion milled cones while preventing deposition on the recessed W fibers. There are several factors that make this approach highly attractive:

1. The ion milled cones appear to be symmetrical with the cone axis perpendicular to the matrix surface when the fibers are perpendicular to the matrix structure. This allows the vapor deposited dielectric and anode to have a circular opening producing symmetrical field gradients.
2. Processing is rapid and contamination may be kept to a minimum. Ion milling required only an hour to produce cones of sufficient size and both vapor depositions could be performed in three hours. Since all operations are vacuum processes, the entire structure could be fabricated sequentially in one vacuum chamber without exposing the sample to air.



(a)



(b)

Figure 1. Schematics of (a) Cones Produced in UO<sub>2</sub>-W Composites by Argon Ion Milling and (b) Low Voltage Emitter Structures After Sequential Low Angle Deposition of a Dielectric and a Metal on a Rotating Ion Milled Composite.



3. Large arrays of electron guns may be fabricated which should be applicable to a variety of electron devices.

Cone size is very uniform within each grain but size variation does occur from grain to grain in the composite. However, the variation is small enough that it should be possible to produce a structure that will emit across a high percentage of the surface.

4. Production of a single emitting pin seems feasible by using an ion microprobe to mill a single cone and processing the sandwich structure as described above. By milling cones at desired locations, controlled redundancy could be produced.

A feasibility study for this process has been conducted in the last three months. The cones have been ion milled using argon ion beam, a dielectric thin film of silicon monoxide has been deposited followed by a metallic coating of aluminum as the anode, and observations in the SEM show that holes are retained for electron emission. The remaining step in the feasibility study was to demonstrate that such a structure would emit. This too, has been accomplished and an emission level of  $5\mu\text{A}/\text{cm}^2$  at 15 volts has been documented. Details of the structure fabrication will be presented in the "Ion Milled Structure" section. Work continues on producing a sandwich structure using chemical etching and will be discussed in the "Chemically Etched Structure" section.

#### A. Chemically Etched Structures

Work has continued to fabricate a low voltage structure by

chemical etching techniques. Techniques for sputtering of  $\text{Al}_2\text{O}_3$  and resistance measurements have progressed to the point that constant resistivities have been obtained. It has been thought that the resistivities of the  $\text{Al}_2\text{O}_3$  films may be too high to permit sufficient electron flow through the film to achieve etched holes in the tungsten film anode. Therefore, studies are being conducted on sputtering thin films of  $\text{Al}_x\text{Cr}_{2-x}\text{O}_3$  with  $x$  varying from 1.5 to 2.0 to obtain films ranging in resistivity from approximately  $10^8$  to  $10^{16}$  ohm-cm. Resistivities for alumina-chromia composites have been reported on bulk samples to temperatures as low as  $400^\circ\text{C}$ . Extrapolation of these values to room temperature indicate that  $10^8$  to  $10^{16}$  ohm-cm resistivities should be obtained but no literature values have been found for thin film alumina-chromia compositions. The dielectric strength of these films are also being measured since it is necessary to know the maximum voltage the films can withstand during emission testing. Targets of alumina-chromia have been fabricated for the sputterer and one composition,  $\text{Al}_{1.75}\text{Cr}_{0.25}\text{O}_3$  has been successfully tested.

#### B. Ion Milled Structures

A disc of  $\text{UO}_2$ -W composite, sample R-3, was copper brazed to an 1/8 inch diameter molybdenum cylinder. After polishing to a one micron diamond finish, R-3 was ion milled in a 100 A, 9 kv argon ion beam for 80 minutes. The W fibers were parallel to the ion beam and the sample was rotated at 60 rpm about the axis of the molybdenum cylinder.

Scanning electron micrographs showed fairly uniform sized cones over a high percentage of the sample surface.

Silicon monoxide was conventionally vapor deposited at a  $12^\circ$  angle to the sample surface with the sample rotating at 30 rpm, Figure 2. As monitored by a piezoelectric microbalance,  $7000\text{\AA}$  were deposited perpendicular to the vapor beam. At an angle of  $12^\circ$ , this calculated to be  $1460\text{\AA}$  of SiO perpendicular to the sample surface. A layer of aluminum  $750\text{\AA}$  in thickness was deposited over the SiO to serve as an anode. It was impossible to see where the coatings stopped on the cone walls. This means either the boundary between the coating and the  $\text{UO}_2$  cone was very smooth or the depositions extended to the bottom of the cones. Some areas were found where there was definitely a deposit on the fibers. In future depositions, the angle between the vapor beam and the composite surface should be decreased.

The sample was emission tested with the electrical conditions specified in Figure 3. After evacuating the sample to  $1 \times 10^{-9}$  torr, the sample did emit at modest current levels. A measurable current ( $\approx 10^{-11}$  Amps) was produced at 13 volts and a few nanoamps were measured at 30 volts. Emission was confirmed by reversing anode-cathode polarity and by diverting the electron beam with a large magnet, both of which eliminated emission current at the collector. The current was very erratic, but it was encouraging that the sample emitted at this level for eight days. Leakage current across the SiO at 50 volts was 1-2 mA for the first few days of

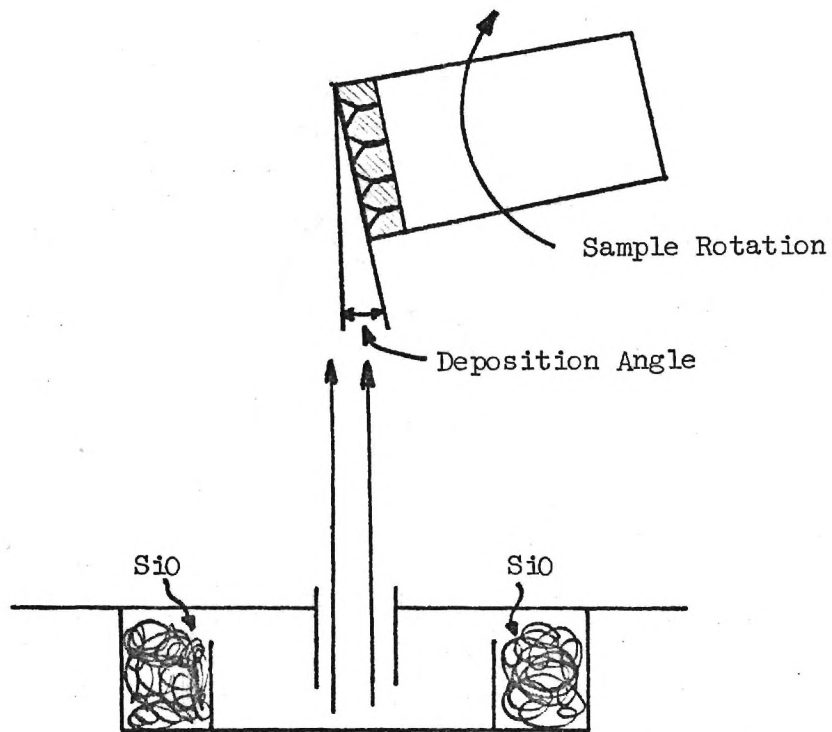


Figure 2. Schematic of Low Angle Deposition of SiO<sub>2</sub> on a Rotating Ion Milled UO<sub>2</sub>-W Composite.

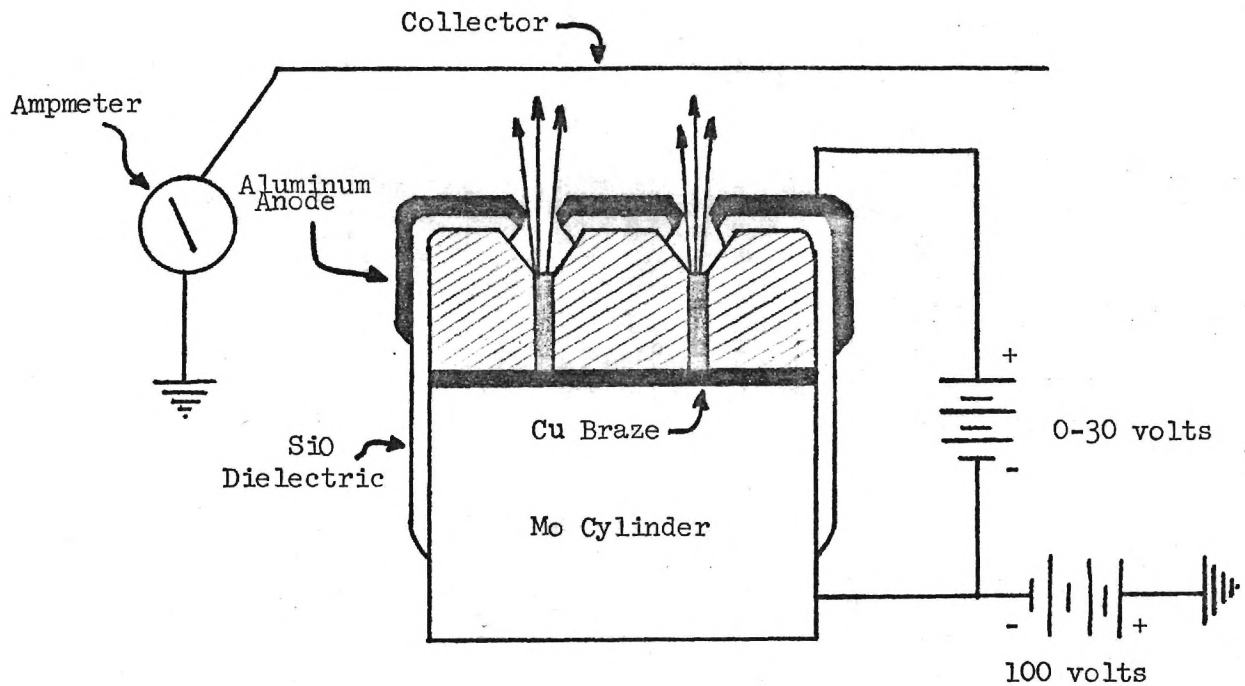


Figure 3. Schematic of Electrical Test Conditions for Sample R-3 Which Produced Field Emission at 13 Volts.

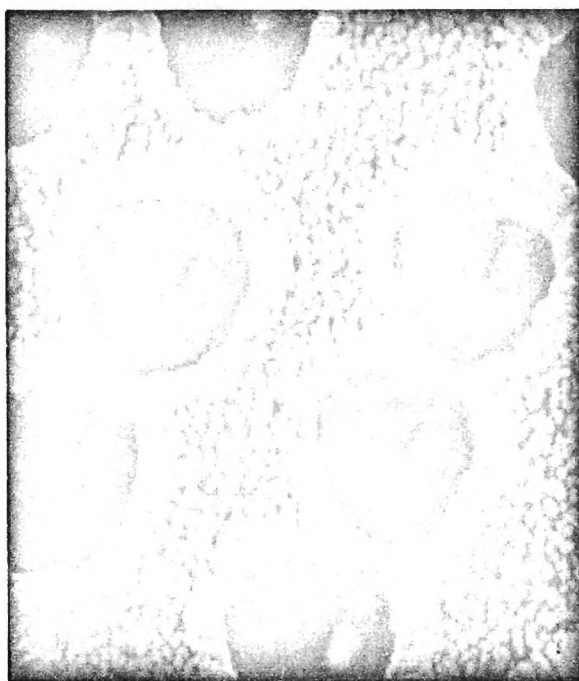
of testing and had risen to 8mA by the 8th day. Emission was terminated at this point to test another sample.

A second low voltage structure, Sample T, was processed for emission testing. Ion milling parameters were identical to R-3. SEM observation showed that these ion milled cones were more uniform than for R-3.

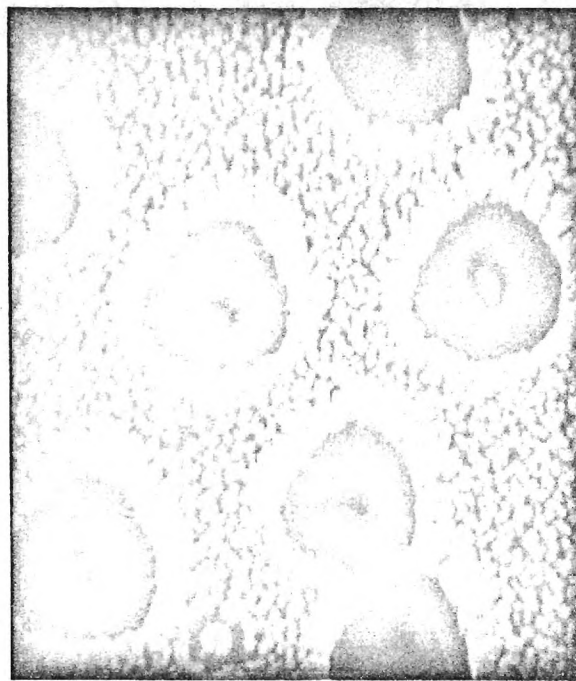
Dielectric and anode coatings were deposited at  $7^{\circ}$  angle to the  $\text{UO}_2$ -W composite surface as compared to  $12^{\circ}$  for R-3. Silicon monoxide was deposited to a thickness of 2500 Å and gold was deposited over the SiO to 300 Å thickness. SEM examination was very encouraging, Figure 4. Reducing the deposition angle produced coatings that were confined to the outside edges of the ion milled cones. The coatings tapered toward the cone center as the film thickness increased resulting in holes centered about each fiber and smaller in diameter than the underlying cones. The grainy texture of the surface and the columnar appearance of the hole walls suggested very porous coatings.

Emission testing of T was identical to sample R-3. Silver paste, again used for establishing electrical contact to the anode, covered approximately 50% of the emitting surface. The sample first emitted at a potential of 8 volts ( $\approx 10^{-11}$  A) and reached a current of 0.2  $\mu\text{A}$  at 15 volts. Emission continued at this level for 18 hours at which time the anode lead wire shorted to the moly cylinder base and melted, terminating emission.

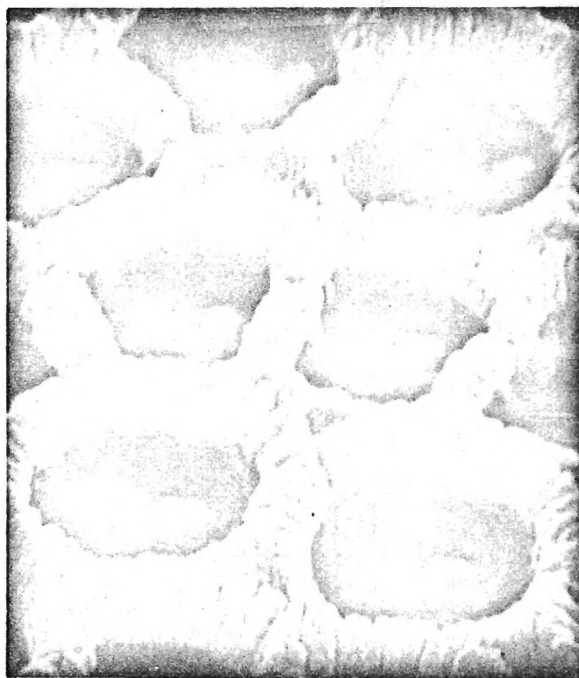




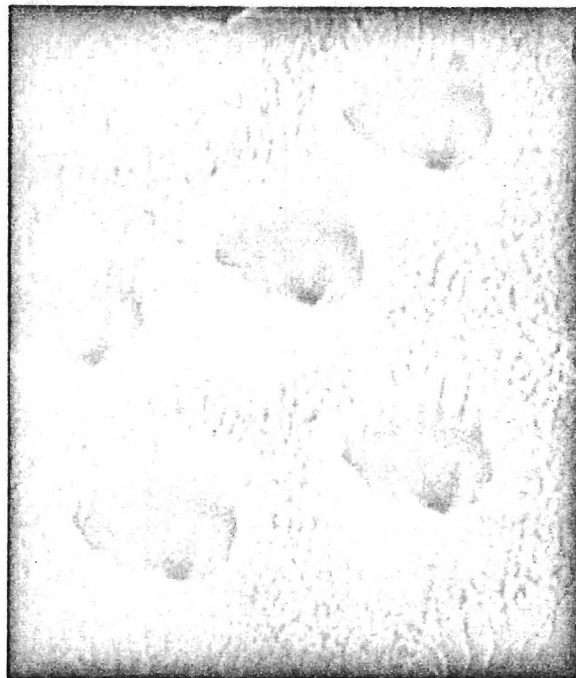
(a)



(b)



(c)



(d)

Figure 4. Scanning Electron Micrograph of Sample T Showing 2500 Å of SiO and 300 Å of Au Vapor Deposited at a 7° Angle on Argon Ion Milled UO<sub>2</sub>-W; (a) and (b) at Viewing Angle of 20° and (c) and (d) at 45°, 12,000x.

Leakage current through the dielectric was 8 mA initially at 15 volts and had increased to 80 mA shortly before the anode lead wire melted.

The emitting surface area was estimated to be  $0.04 \text{ cm}^2$  and a fiber density of  $12 \times 10^6 / \text{cm}^2$  was calculated from Figure 4. Thus, at the  $2 \times 10^{-7}$  A emission current at 15 volts, sample T was emitting at  $4 \times 10^{-6} \text{ A/cm}^2$  and an average of  $4 \times 10^{-13}$  /pin. Since there is no way of knowing how many pins were emitting, emission from an individual pin may have been much greater. It was also disappointing that leakage current was so high, since emission current might have increased orders of magnitude at increased potential.

### III. Electron Emission Performance

During this report period CBS Sample No. 6 was installed in the test diode for the initial investigation of the high temperature processing ( $> 1000^\circ\text{C}$ ) available with a platinum brazed sample. For this test the improvement and modification of the test diode noted in the August Monthly Report were utilized. After the low temperature bake-out was accomplished with heating tapes, the temperature of the sample was slowly raised to approximately  $1300^\circ\text{C}$  over a two day period. Care was taken to heat both the cathode as well as the new anode fabricated from the arch-fused Mo. Immediately after processing was terminated an anode potential was applied to produce a gross emission current of 150  $\mu\text{A}$ , which resulted in a current per pin of about 3nA since the sample contained 50,000 pins. During the 10 minute period required to raise

the emission current to this value the noise that was initially  $\pm 10\%$  rapidly stabilized at  $\pm 2\%$ . These emission characteristics were significantly better than ever observed during the initial "turn-on" of any previous emitter sample.

During the next several hours of testing there was some spurious emission behavior attributed to the cooling of the diode assembly or some electronic problems (an input voltage regulator was employed during this period and its behavior is suspect since it failed the following day). After operating for 24 hours the noise characteristics were continuously monitored and the current fluctuations were not detectable on the strip chart recorder. During the following 6 days short-term fluctuations in emission current were not detectable; occasionally during long-term periods there was a small apparent increase or decrease in the gross emission. It is uncertain whether these small changes reflect emitter stability or the electronic stability of the associated power supply, electrometer, or recorder

The ion pump "tripped-out" sometime during the 7th day of emission testing when a student inadvertently disconnected a power line. The pressure rose in the diode and since it was operating at 20 kv a major arch occurred across the diode which destroyed the sample. On disassembly the emitter was found in fragments at the bottom of the glass tube.

The significant result from this experiment, CBS-6, was the value of the high temperature ( $1400^{\circ}\text{C}$ ) processing. This treatment

resulted in an emitter which performed, after the initial few minutes of activation, with far greater stability than had been previously observed.

The second platinum brazed sample designated CBS-7 was also tested this report period. The sample selection for this experiment was limited since only one additional composite sample had been successfully platinum brazed to a Mo cathode structure. The sample was processed to  $1400^{\circ}\text{C}$  over a 2 day heating cycle but when the anode voltage was applied almost no emission current was obtained. The cause of this poor performance was evident on post-emission examination. The sample contained greater than 30 million pins/cm<sup>2</sup> and, as predicted by theory, the high pin density and close pin spacing behaved as a parallel plate geometry. The only positive result obtained from this experiment was the influence of heating on the shape of the pin tips. After processing to  $1400^{\circ}\text{C}$  the pin tips were well rounded and, in some instances, slightly larger in diameter than the pin itself. This suggests that with processing at a  $1000^{\circ}\text{C}$  and above it may be impossible to retain the pointed tip geometry with the composite emitters.

A third emission tested sample, CBS-8, was also completed this report period. In this experiment a  $\text{UO}_2$ -W composite with an excellent pin array and pointed tips was tested. The unique feature of this experiment was the incorporation of a polished 0.003" diameter platinum wire located 0.10" above the emitter. In this geometry approximately 500 pins were located directly under the

wire anode. The objective was to investigate the maximum current-per-pin characteristics of the emitter. Since the cathode and anode were constructed from a variety of different materials, including a sample which was copper brazed, no processing other than the low temperature bake-out was employed prior to testing. The following is a brief chromological description of the emission testing CBS-8.

The sample was initially "turned-on" at an applied voltage of 4.5 kv yielding a gross current of  $8 \mu\text{A}$  with noise characteristics of  $\pm 15\%$ . The voltage was slowly increased over a 3 hour period to approximately 8 kv with a total current of  $150 \mu\text{A}$ . The noise characteristics decreased to  $\pm 2\%$  over this period even though the system pressure had increased to  $10^{-4}$  torr.

The following day the applied voltage was increased to 10.3 kv with a resulting current of  $300 \mu\text{A}$ . (Incidentally this data was recorded the day of the visit of Jim Greed and Holgar Luther from CBS.) This current was the limit of the "Ransburg" power supply and the sample was switched to the 50 kv U.S. Army Missile Command power supply. The voltage was next increased to 11.9 kv with a total current of  $500 \mu\text{A}$  when interelectrode arching was observed. The voltage was decreased to 11 kv producing  $200 \mu\text{A}$  and the current stability was very poor. Switching back to the "Ransburg" power supply and operating at similar V-I conditions resulted in a very stable emission current for a period of 4 days. The reason for this difference in noise characteristics between the two power supplies is uncertain at present.



Since the objective of this experiment was to operate at 500  $\mu$ A the larger power supply was returned to the emission circuit and the sample was subsequently run an additional 8 hours at 500  $\mu$ A. Although the contribution to the total emission current from pins not directly under the wire anode is uncertain, it appears reasonable to speculate that 50% of the total current is from 500 pins (since emission decreases rapidly with increased spacing). Thus these data show a current-per-pin capability from the oxide-metal composites of 500 nA. Post emission examination of this sample has not been completed.

On November 21 CBS Sample No. 3 was removed from testing to make the chamber available for the low-voltage emission experiments. This sample has run almost continuously for 3700 hours. Prior to removal the noise was monitored for 12 hours with no detectable current variations on a strip chart recorder. Post emission examination will be reported next month.

#### IV. Financial Report

The estimated expenditure noted for the month of October in the August Monthly Report was incorrect. At the end of November the contract funds remaining are approximately \$3500. These funds will largely be used for SEM time during the remaining contract period (proposed extension through March 31, 1975).

MONTHLY TECHNICAL STATUS REPORT

1 December 1974 - 1 March 1975

HIGH STABILITY LONG LIFE FIELD EMISSION ELECTRON  
SOURCE WITH A HIGH BRIGHTNESS

Subcontract for AF Prime Contract  
F08606-73-C-0028

CBS Laboratories No. 52501

School of Ceramic Engineering  
Georgia Institute of Technology  
Atlanta, Georgia 30332

# MONTHLY TECHNICAL STATUS REPORT

on

## DEVELOPMENT OF A HIGH STABILITY LONG-LIFE FIELD EMISSION ELECTRON SOURCE WITH A HIGH BRIGHTNESS

CBS LABORATORIES NO. 52501

This report is the combined 13th, 14th, and 15th Monthly Status Report, which covers the period from 1 December 1974 to 1 March 1975. The report is divided into several sections covering different aspects of the research program.

### I. Preparation of Arrays Having a Small Number of Pins.

A series of  $\text{UO}_2$ -W composite samples after processing to form typical emitter geometries were installed in the SEM for the "mechanical shaving" with the "Micro-Manipulator" to form single or few pin structures. A variety of different procedures were tried and the following description summarizes the problems and the "best" techniques available at present.

It was initially established that samples with shorter pins (5  $\mu\text{m}$  instead of the typical 10  $\mu\text{m}$  length) were easier to handle in the SEM and the latter shaving trials were all made with pins of this shorter length. Before installation in the SEM, the samples were "hand-shaved" using a scalpel with the binocular microscope to reduce the number of pins to a level that would require a minimum amount of time on the SEM for final sample preparation. The micro-manipulator, as received, did not possess fine enough vernier controls, as the minimum travel in any one direction was approximately 100  $\mu\text{m}$ .

A mechanical reduction of 50-to-1 was attached to the controls to increase the motion sensitivity.

With the aid of mirrors, external light sources and accurate manual positioning, the samples were placed in the SEM and the manipulator probe was moved into a position close to the sample. After evacuation the probe point was centered on the viewing screen, and using the stage controls of the SEM, the sample was brought into contact with the probe. The probe tips were made by electrochemically etching 5 mill tungsten wire to an acceptably fine point.

The sample surface was slowly scanned at 500 to 1000X and the topography carefully noted for selection of an appropriate site for emitter preparation. Once a site was selected, the surrounding pins were removed by moving the sample back and forth under the probe, rotating slightly between each sweep. The emitter site itself consisting of several thousand pins was next sectioned by cutting perpendicular swaths to create separate clusters of pins. Each section was then reduced to several pins. Out of the 5 to 8 clusters per sample, only one or two were typically suited for further use because other pins were often pushed into the clusters during the previous shaving passes.

Each of the few pin regions were carefully studied to determine the proper angle of attack for the easiest elimination of the extraneous fibers to leave but a single pin. This last step was most sensitive since the fibers are separated only 1.0 to 1.5  $\mu\text{m}$  and the 5  $\mu\text{m}$  long fibers that were

previously cut down tend to be attracted to the probe by electrostatic forces.

Figure 1 shows the successful shaving to form the single pin geometry.

Unfortunately ultrasonic cleaning in a 2% solution of TSPP occasionally removed some standing pins and reduced the output of emitters.

With the experience gained during these shaving trials with the "Micro-Manipulator" it is anticipated it will be possible to document the emission characteristics of a single free standing pin. Obviously further development work both with the shaving process and the insulating or conducting layer needed to cover the adjacent flush pins is required to meet this objective.



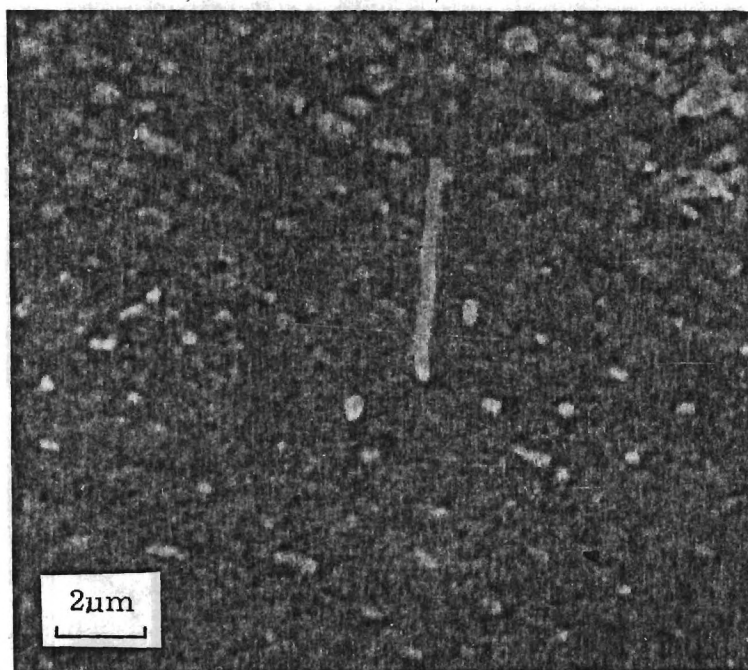


Figure 1. Oxide-Metal Composite of  $\text{UO}_2$ -W that has been Micromachined in a Scanning Electron Microscope to Leave a Single Free Standing Tungsten Fiber, 6,000X.

## II. Low Voltage Field Emitters

Since the last monthly work has continued to produce a low voltage sandwich structure based on recessing the tungsten fibers in ion milled cones of  $\text{UO}_2$  and low angle deposition of dielectric and anode coatings. Work on sandwich structures by chemical etching of selectively anodized tungsten thin films has been discontinued due to the progress made with ion milled structures. Efforts in the last three months have been directed toward controlling the geometry of ion milled cones, achieving free standing tungsten fibers in ion milled cones, and further emission testing of low voltage structures.

### A. Geometry of Ion Milled Cones

One  $\text{UO}_2$ -W composite, sample Q, was noted to ion mill producing uniform cones over most of its surface, Figure 2 and it was selected for investigation of the effect of ion energy on cone geometry. Sample Q was processed by brazing to a 0.125" diameter molybdenum post and polishing to a one micron diamond finish. All ion milling was carried out at an argon beam current of 100  $\mu\text{a}$  as measured at the sample with the sample grounded for a duration of one hour. The sample was milled using ion acceleration voltages of 6.0 to 12.0 KV with the sample rotating at 60 rpm. After photographing in the SEM, the sample was repolished with one micron diamond paste to remove the previously milled surface.

The angle of the three cones with respect to the  $\text{UO}_2$  surface was calculated from SEM photographs for each ion energy investigated, Table 1.

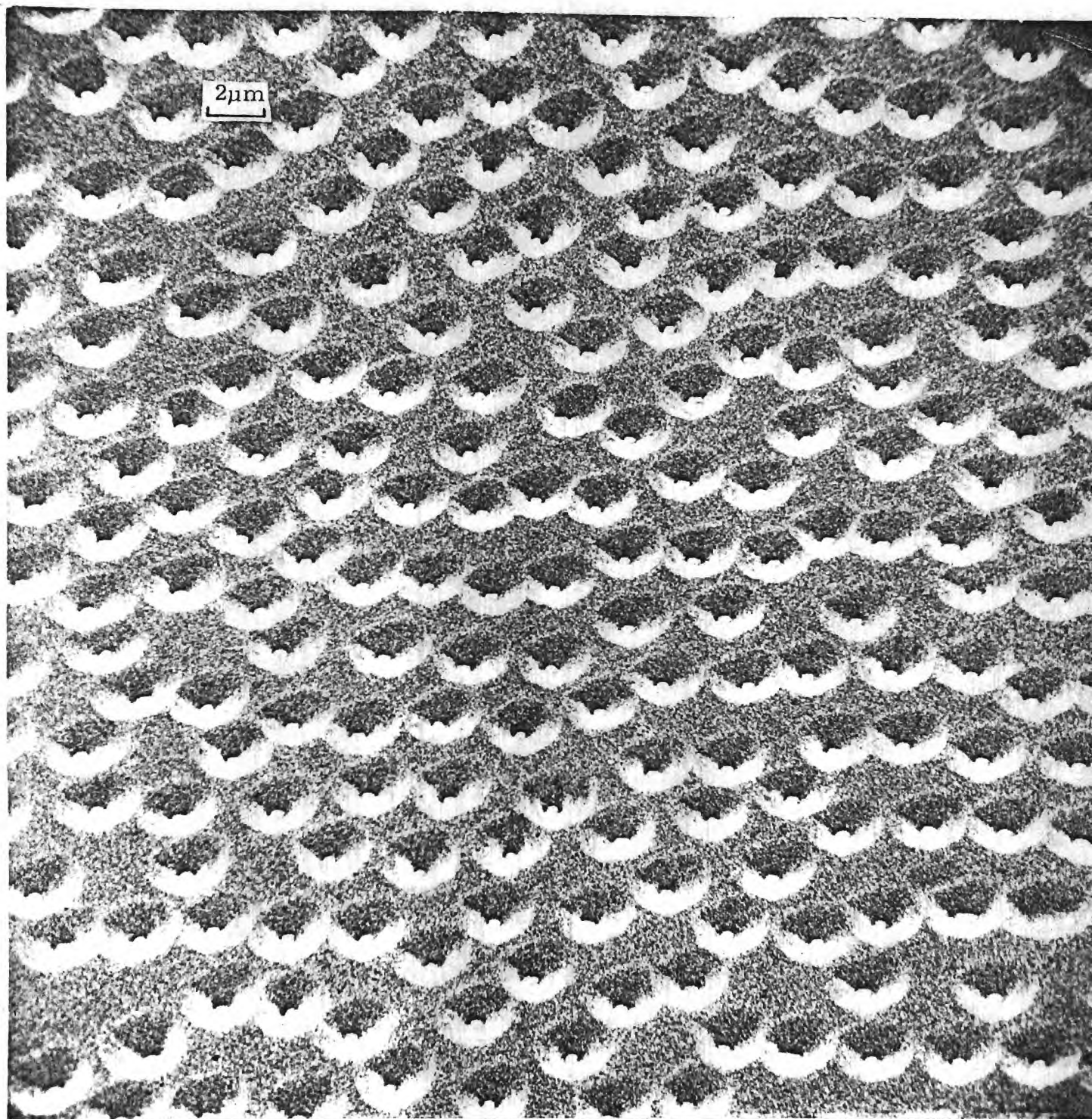


Figure 2. Scanning Electron Micrograph of Sample Q, Ion Milled at 9 KV and 50  $\mu$  a for One Hour, 4,400X.

Table I. Effect of Ion Energy of Geometry  
of Ion Milled Cones in  $\text{UO}_2$ -W  
Composites, Sample Q.

Sample	Ion Energy (KV)	Cone Angle (degrees)	Average Width of Cone at Surface ( $\mu\text{m}$ )	Cone Depth ( $\mu\text{m}$ )
25Q	6.0	$16.4 \pm 2.8^*$	1.87	0.21
32Q	7.5	$23.2 \pm 0.6^*$	3.00	0.59
23Q	9.0	$27.9 \pm 1.5^*$	2.78	0.64
31Q	10.5	$29.2 \pm 0.3^*$	3.02	0.81
28Q	12.0	$39.9 \pm 0.5^*$	2.45	0.66

\*Average of 3 cones. Error is mean deviation.

As can be seen in Figure 3, the cone angle with respect to the  $\text{UO}_2$  surface increased with voltage ranging for  $16^\circ$  at 6 KV to  $40^\circ$  at 12KV. The scatter in the data, Figure 4, is probably due largely to the cones axis not being perpendicular to the  $\text{UO}_2$  surface and to nonsymmetrical cones as these two assumptions must be made to calculate cone angle. Using these two assumptions, the cone angle,  $\phi$ , was calculated knowing the viewing angle in the SEM,  $\theta$ , and the lengths  $l_1$  and  $l_3$ , Figure 5.

Referring to Figure 5;

$\phi$  = cone angle with respect to the  $\text{UO}_2$  surface,

$\theta$  = viewing angle in the SEM,

$r$  = fiber radius,

$d$  = cone depth,

and the other lengths are labeled as shown. Cone angle was calculated as follows.

$$\tan \phi = d/l_5 \quad (1)$$

$$d = l_6 \tan \theta \quad (2)$$

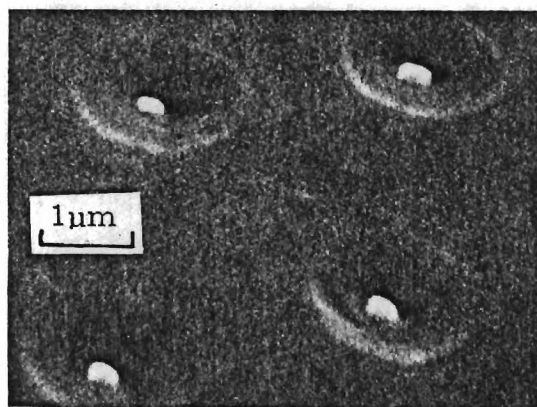
$$l_6 = l_7 - l_5 \quad (3)$$

Combining 1, 2, and 3,

$$\tan \phi = \frac{l_7 - l_5}{l_5} \cdot \tan \theta = \left( \frac{l_7}{l_5} - 1 \right) \cdot \tan \theta \quad (4)$$

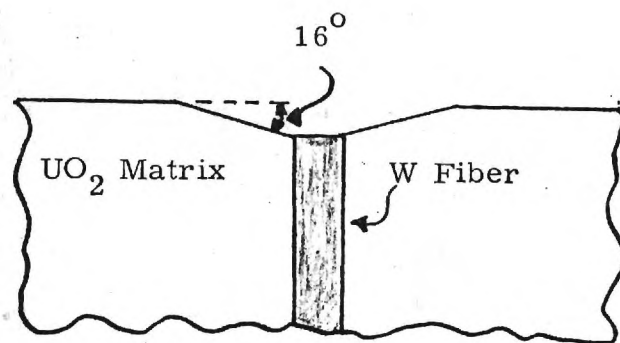
$$l_7 = l_3 / \sin \theta \quad (5)$$



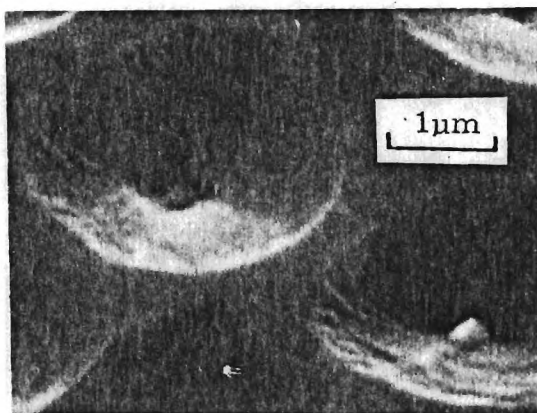


6.0 KV; 12,100X

(a)

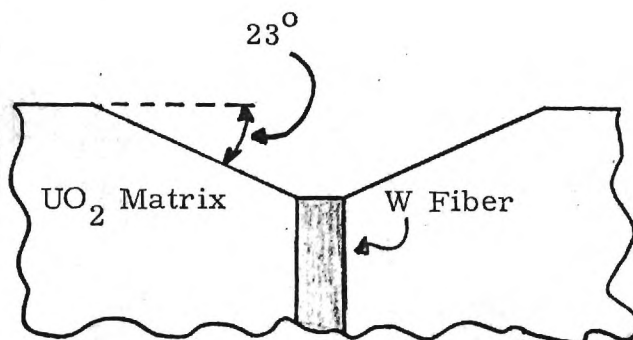


Scale 20,000X

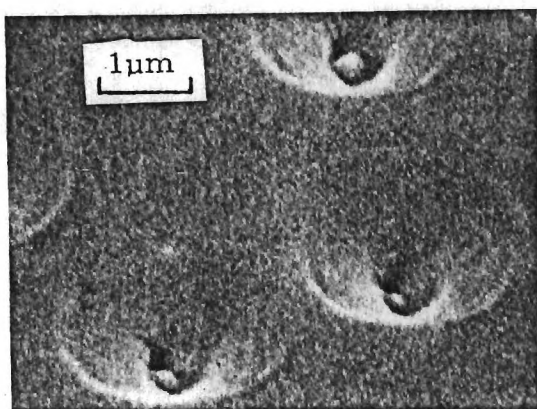


7.5 KV; 13,800X

(b)

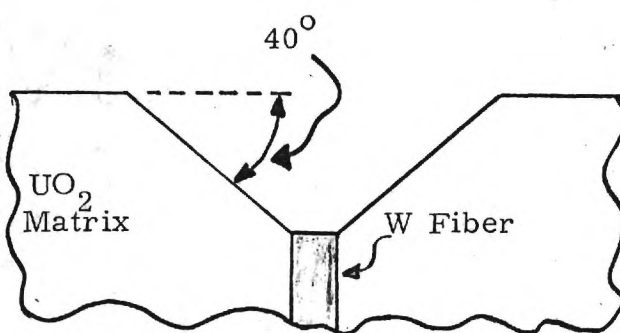


Scale 20,000X



12.0 KV; 12,100X

(c)



Scale 20,000X

Figure 3. Scanning Electron Micrographs of  $\text{UO}_2$ -W Composite, Sample Q, Showing Effect of Ion Energy on Angle of Ion Milled Cones. (All SEM's at Viewing Angle of  $45^\circ$ )

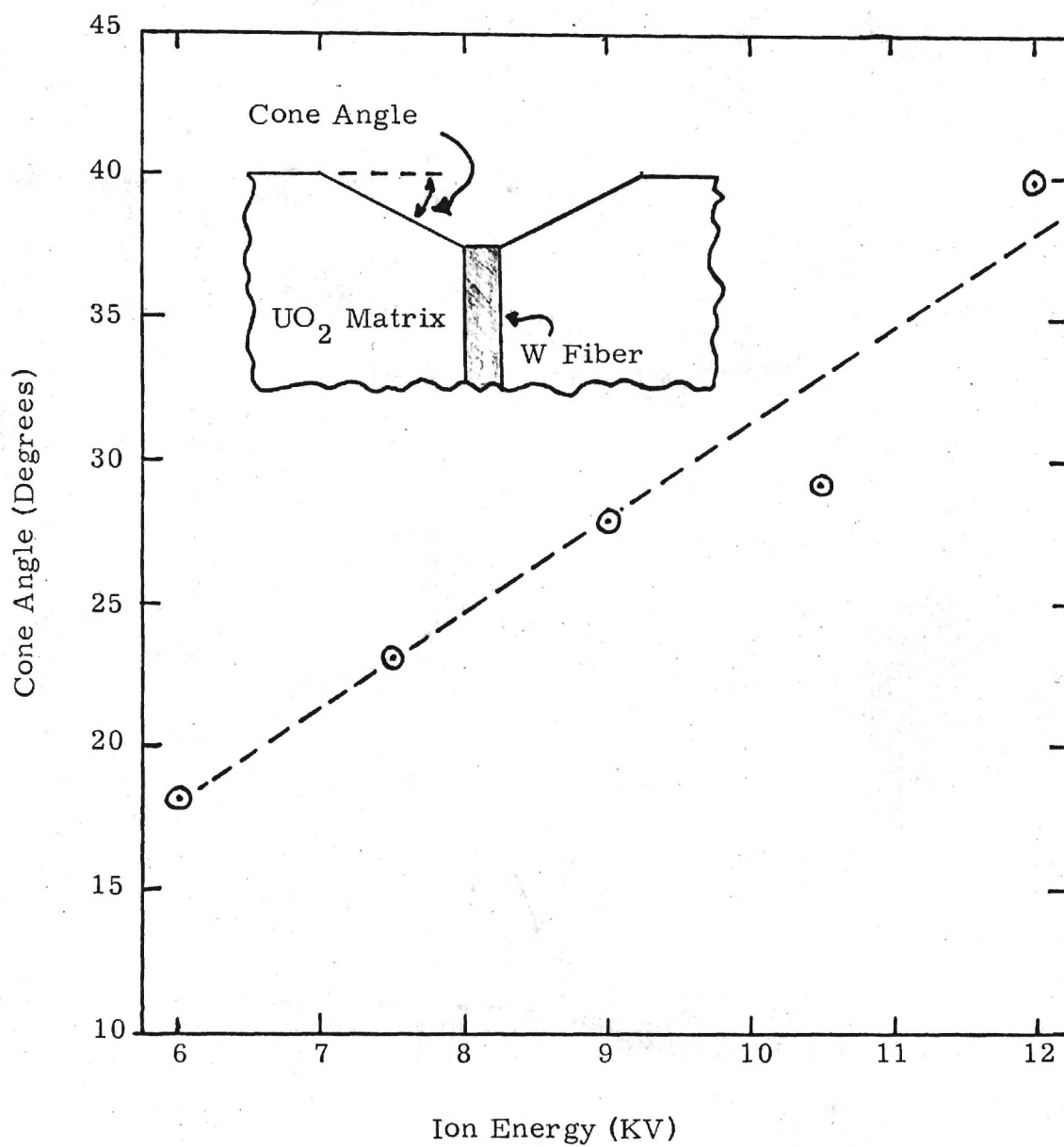


Figure 4. Effect of Ion Energy on Angle of Recessed Cones Ion Milled in UO<sub>2</sub>-W Composite, Sample Q.

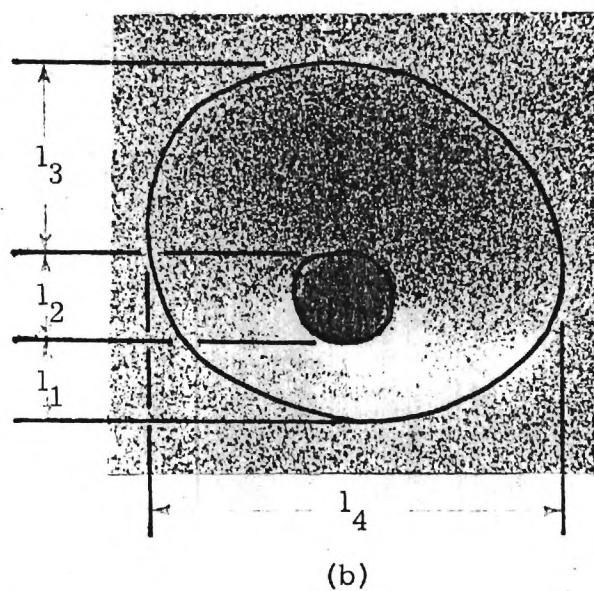
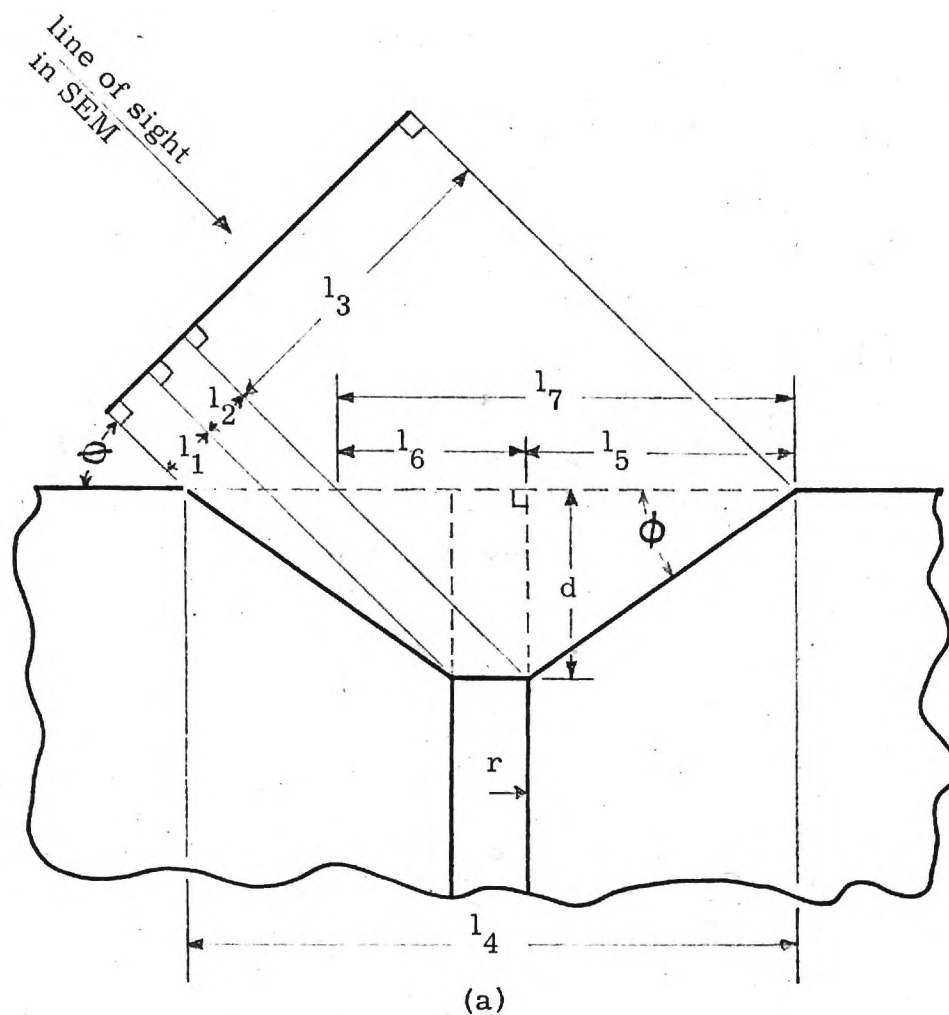


Figure 5 . Parameters for Calculation of Ion Milled Cone Geometry, Shown (a) in a Side View, and (b) as Measured from a SEM Photograph

$$l_5 = \frac{l_4}{2} - r = \frac{l_4}{2} - \frac{l_2}{2 \sin \Theta} \quad (6)$$

$$l_4 = \frac{l_1 + l_2 + l_3}{\sin \Theta} \quad (7)$$

Combining (5), (6) and (7) and rearranging,

$$\frac{l_7}{l_5} = \frac{l_3}{\sin \Theta} \bigg/ \frac{l_3 + l_1}{2 \sin \Theta} \quad (8)$$

$$\text{or } \frac{l_7}{l_5} = \frac{2 l_3}{l_3 + l_1} \quad (9)$$

Combining (4) and (9) and rearranging,

$$\tan \phi = \left( \frac{l_3 - l_1}{l_3 + l_1} \right) \cdot \tan \Theta \quad (10)$$

Since for all photos  $\Theta = 45^\circ$ ,  $\tan \Theta = 1$ , and

$$\tan \phi = \frac{l_3 - l_1}{l_3 + l_1} \quad (11)$$

Cone depth, d, was calculated from,

$$d = \left( \frac{l_3 - l_1}{2 \cos \Theta} \right) \cdot \text{SEM Magnification} \quad (12)$$

and cone width was measured directly from the scanning electron micrographs.

Certainly, there are many sources of error in describing the cones by this method but it does show that the geometry of the cone may be

controlled on an individual sample to hopefully optimize emission from low voltage emitters at a later date. For now, it is important to know cone geometry to determine the maximum angles at which dielectric and anode coatings may be deposited. Other parameters such as fiber density, ion current, and milling time, probably also have an effect on the geometry of ion milled cones in  $\text{UO}_2$ -W composites and they offer areas of investigation in the future.

#### B. Free Standing Recessed Fibers

To provide field enhancement for low voltage emission, it is desirable to produce fibers that are free standing in the ion milled cones and are recessed below the surface of the  $\text{UO}_2$ . Two techniques to produce free standing recessed pins were attempted. In the first, a chemical etch that had been shown previously to produce pointed fibers in holes with the pin tips in the plane of the  $\text{UO}_2$  surface was employed after samples had been ion milled to geometries similar to Figure 2.

Using the following etch;

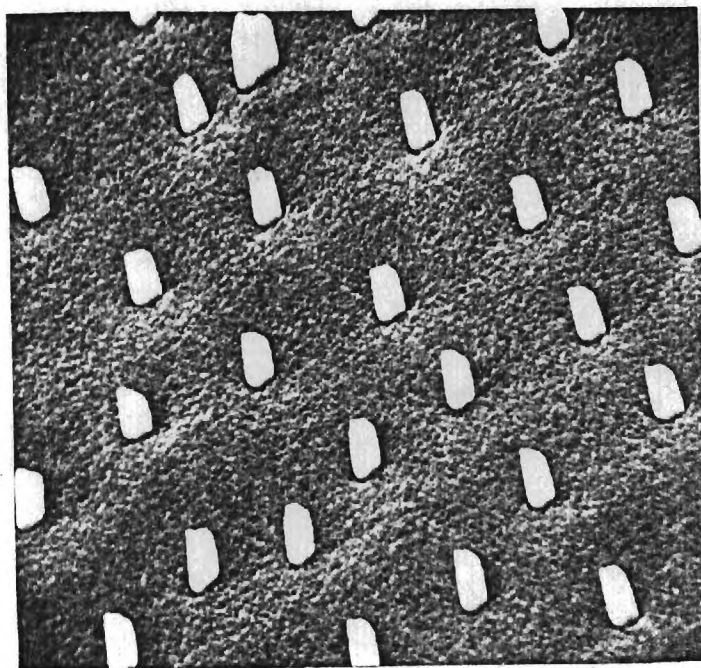
<u>Acid</u>	<u>Volume Ratio</u>
Glacial Acetic	10
Saturated $\text{CrO}_3$	12
Conc. $\text{HNO}_3$	7
Conc. HF	10

both ion milled and polished samples were etched at  $0^\circ\text{C}$  for 5, 10, 20, and 30 minutes. At all times, the ion milled and polished samples looked almost identical.

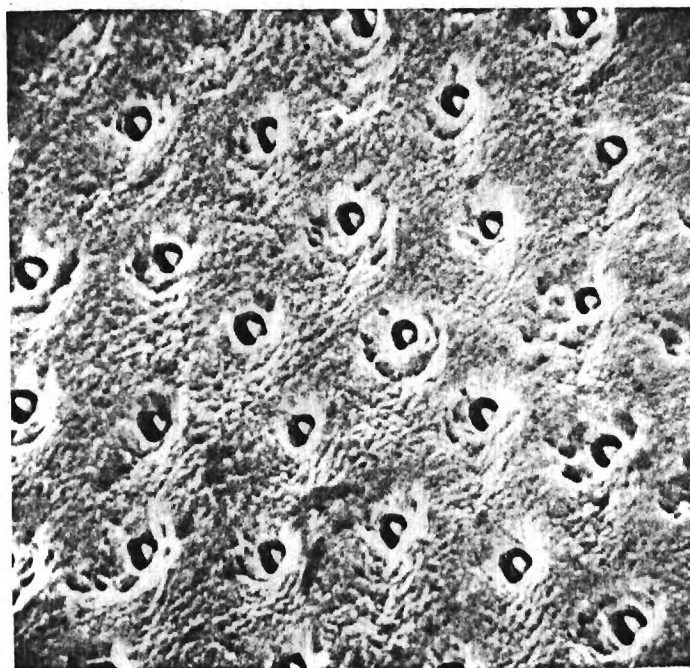


Apparently, the etch attacks the  $\text{UO}_2$  matrix first leaving free standing tungsten fibers and later attacks the fibers. At five minutes etching time, the matrix had been etched to the extent that the ion milled cones had been removed, Figure 6. After 30 minutes the fiber tips were pointed and in the plane of the matrix with the matrix dissolved surrounding the fibers, Figure 6. Thus, due to the etching sequence, chemical etching after ion milling holds little promise of producing free standing recessed pins but it might be possible to ion mill lightly after chemical etching to recess the already free standing fibers in holes.

The second technique, consisted of heating platinum brazed, ion milled samples to  $1500^\circ\text{C}$  for 30 and 60 minutes in  $\text{H}_2$ . After 30 minutes a slight rounding of the fibers in the recessed cones was seen and after 60 minutes the fibers were protruding from the cone base, Figure 7. The 60 minute sample, A-4, was coated and emission tested as described in the next section. The protrusion length seen in Figure 7(b) was greater than expected because, if the cylindrical end of a fiber were converted to a hemisphere the fiber would be extended  $1/3$  of a fiber radius above the original fiber or approximately 500 to 600 Å. As can be seen in Figure 7(b), the lengthening of the fibers was much greater than  $1/3$  r. There is the possibility that due to thermal gradients at the fiber tip what is actually seen is some sort of preferential vapor deposition. This will have to be investigated in the future.

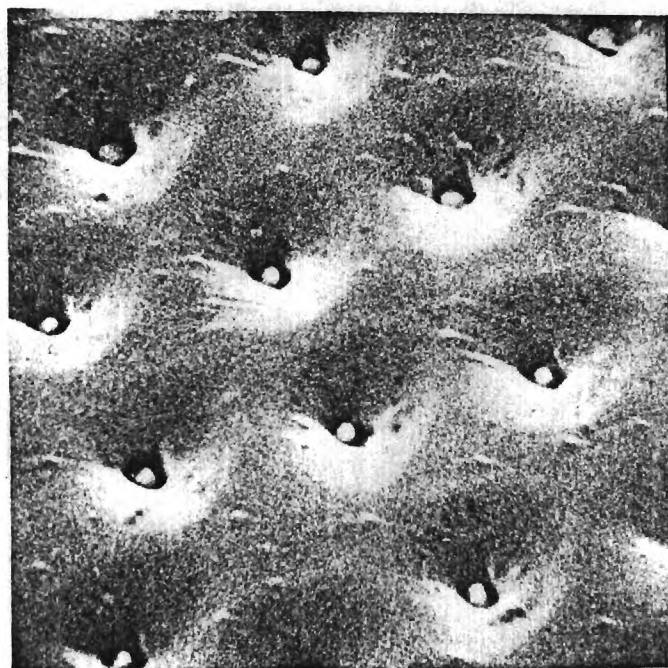


(a)

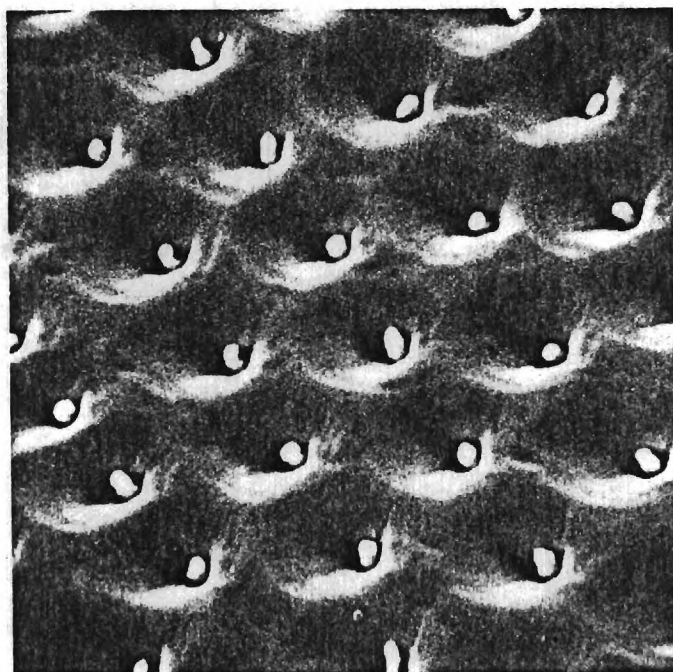


(b)

Figure 6. Scanning Electron Micrographs of  $\text{UO}_2$ -W Composites that have been Ion Milled to 9 KV, and 100  $\mu\text{a}$  for One Hour and Chemically Etched for (a) 5 Minutes and (b) 30 Minutes, (7,000X).



(a)



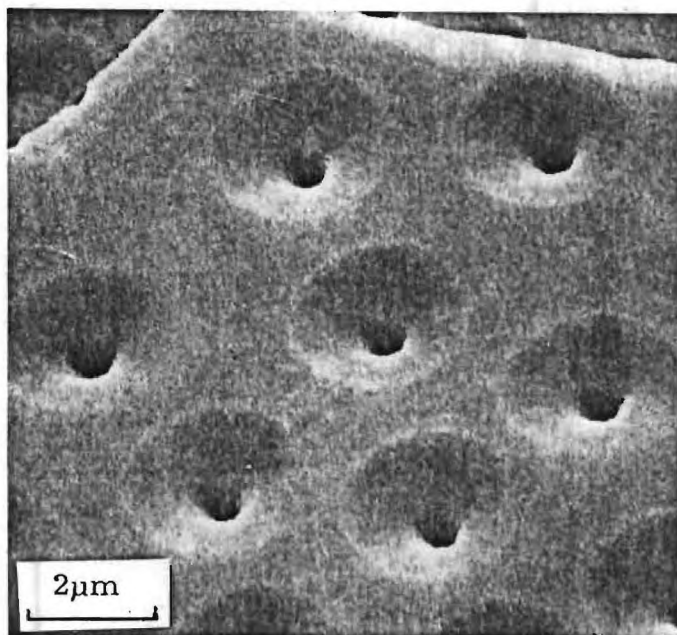
(b)

Figure 7. Scanning Electron Micrographs of Ion Milled  $\text{UO}_2$ -W Composites Show Free Standing Tungsten Fibers Produced by Thermal Treatment at  $1500^\circ\text{C}$  for (a). 30 Minutes (14,000X) and (b) 60 Minutes (7,300X).

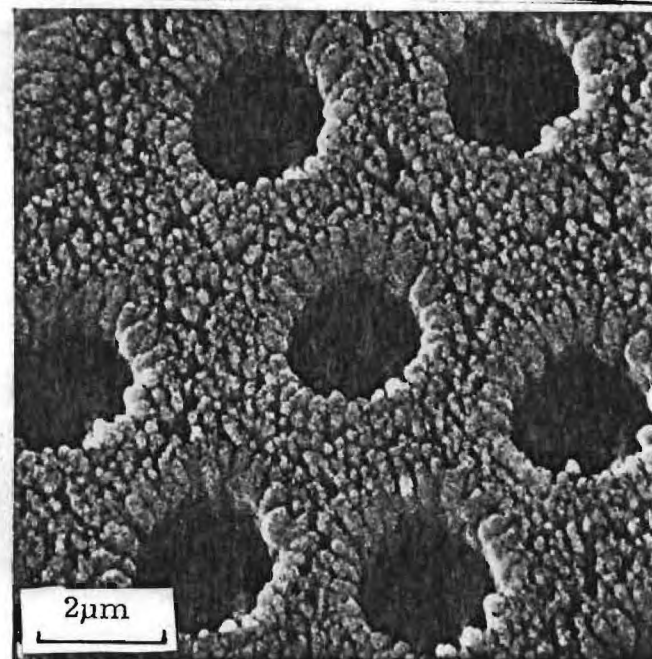
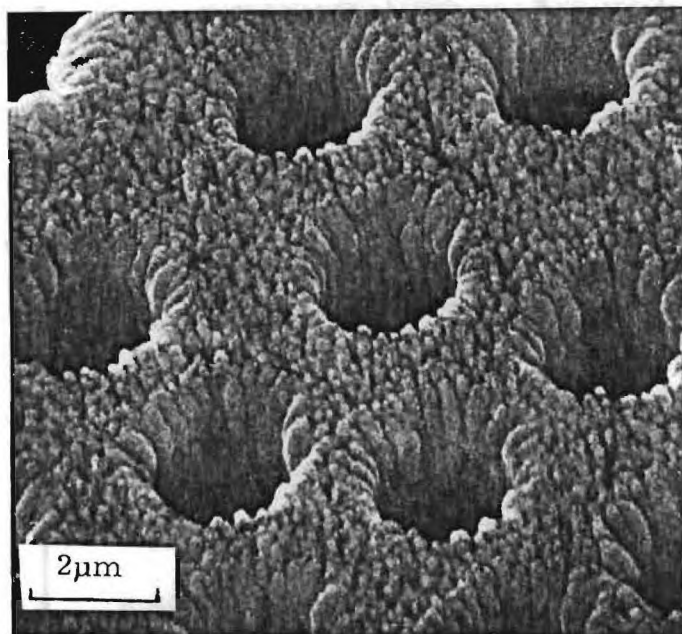
### C. Emission Testing

Two samples have been processed for emission testing since the last report, samples R-9 and A-4. Sample R-9 was ion milled at 9 KV and 100  $\mu$ a for one hour and coated with 5000  $\text{\AA}$  of SiO and 1000  $\text{\AA}$  of Al at a deposition angle of  $7^\circ$ , Figure 8. The coating was porous due to the columnar structure as noted previously and is at least twice as thick as measured during deposition by the vibrating quartz crystal thickness monitor. Also, it was noted during SEM viewing that the fibers had been recessed out of sight in a high percentage of the cones. Emission under conditions described in the last report was low. The sample was biased a negative 500 volts and extraction voltage eventually was as high as 200 volts. Leakage current at 200 volts was only 1.8 mA. Emission was first detected at an extraction potential of 130 volts. The maximum current obtained was  $1 \times 10^{-7}$  amps. This low emission level was probably due to the recessed fibers and the discontinuous film due to low angle deposition.

Sample A-4 as described previously had been thermally treated to produce free standing fibers, Figure 7(b). SiO was deposited on A-4 to a thickness of 2200  $\text{\AA}$  and overcoated with 1200  $\text{\AA}$  of gold with both depositions at a  $10^\circ$  angle. No improvement of the grainy texture was evident, (compare Figure 9 to Figure 8) due to the increased deposition angle. Also the gold anode was apparently shorted to the  $\text{UO}_2$  substrate as distinct light and



Inverted Cones, Ion Milled in a  $\text{UO}_2$  Unidirectionally Solidified Composite. This Milled Composite Served as the Substrate for the Low Voltage Emitter Structure Shown Below and Right. Scanning Electron Micrograph 8,000X  $45^\circ$  Viewing Angle. (Above)



Scanning Electron Micrograph of Ion Milled  $\text{UO}_2$ -W Composite Sequentially Overcoated with 5000 Å of  $\text{SiO}_2$  Dielectric and 1000 Å of Aluminum to Serve as the Anode. Note that the W Fibers are Recessed Below the  $\text{UO}_2$  Matrix. 8,000X; Left,  $45^\circ$  Viewing Angle; Above,  $20^\circ$  Viewing Angle.



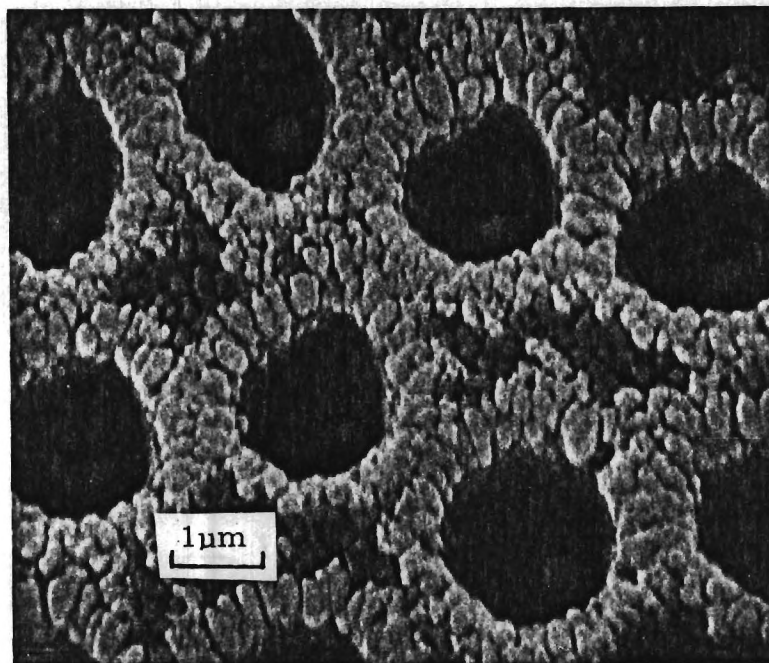
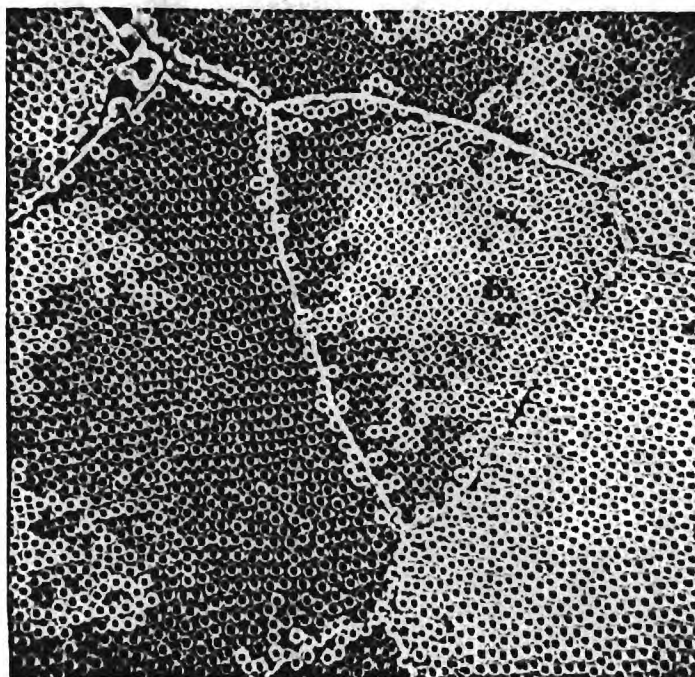
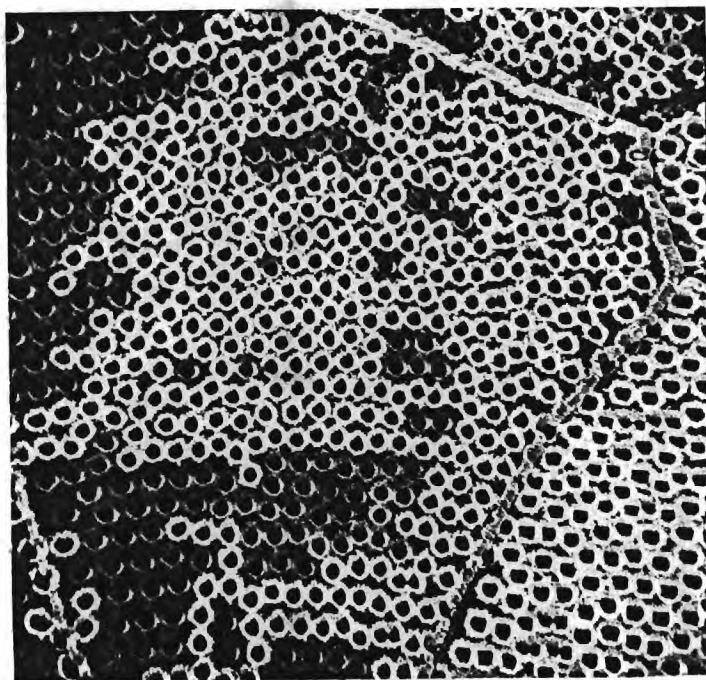


Figure 9. Scanning Electron Micrograph of  $\text{UO}_2$ -W Composite, Low Voltage Emitter Sandwich Structure with Free Standing Tungsten Pins, Sample A-4, 12,100X. This Sample has been Processed by (1) Ion Milling to Produce Recessed Cones, (2) Thermal Treatment to Produce Free Standing Fibers, (3) Vapor Coated with 2,000 Å of  $\text{SiO}_2$  Dielectric, and (4) Vapor Coated with 1,000 Å of Gold to Serve as an Anode.

dark regions were seen in the SEM, Figure 10. On emission testing the sample was shorted. Measurable emission was obtained,  $8 \times 10^{-11}$  amps, at 27 volts for about 30 minutes prior to complete shorting and after that no more than one volt could be maintained on the anode.



(a)



(b)

Figure 10. Scanning Electron Micrographs of Emission Sample A-4 Showing the Surface Anode Shorted to the  $\text{UO}_2$  Substrate, Dark Areas, (a) 550X and (b) 1100X.

### III. Electron Emission Performance

This section describes the post emission examination of CBS Sample No. 3 and No. 8, and the emission performance and post emission analysis of CBS Sample No. 9.

The long-term sample, CBS - No. 3, operated almost continuously for 3,700 hours with a gross current of 10 mA and a current density of greater than  $100 \text{ mA/cm}^2$ . Upon disassembling the test diode the first observation noted was the extensive amount of discoloration present on the ceramic insulators, the water-cooled copper electrodes and on selected areas of the walls of the chamber. The origin of this material is probably Mo vaporized and/or electron-sputtered from the anode. Examination of the anode revealed no gross surface roughness; however, the outline of the sample cell structure was readily discernable in the anode. Scanning electron microscope examination of the  $\text{UO}_2$ -W cathode structure indicated several significant changes:

1. All of the initially sharpened pins were significantly rounded or blunted after operation for 3700 hours, Figure 11.
2. Approximately 10% of the pins were totally destroyed, and this destruction occurred exclusively along the edges of the sample.
3. In one area there were 50 to 100 scattered pins which displayed a flowered-shape growth at their tips which may be attributed to tip vaporization (exploding) prior to the onset of melting.

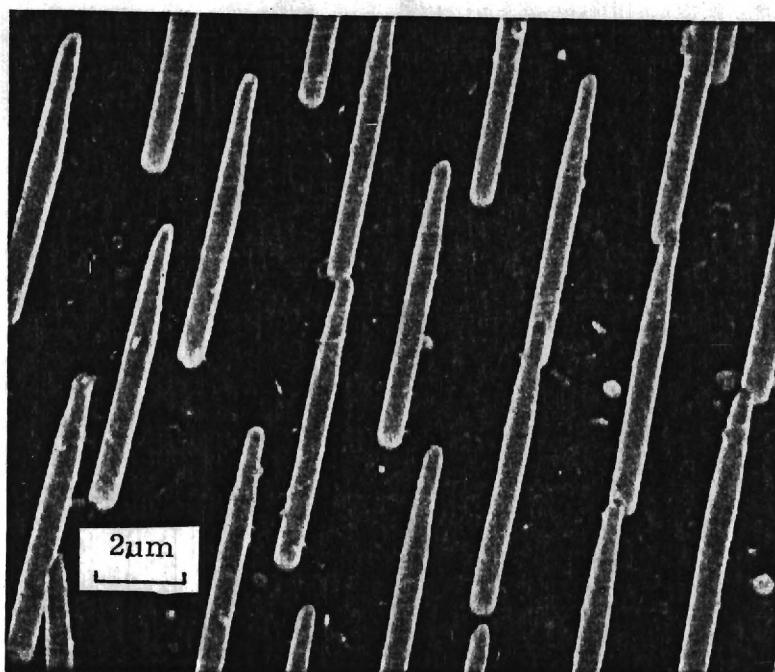


Figure 11. Rounded Emitter Tips from UO<sub>2</sub>-W Composite after 3,700 hours Operation at 10 to 100 nA/pin.



Unfortunately this sample had been used prior to the long-term testing for the high pressure barium treatment designed to enhance the emission characteristics; the damage associated with the burned down pins and exploding tips may have been accumulated during the Ba runs since the following 3,700 hours of testing ran virtually trouble free.

The emission performance of CBS Sample No. 8 was described in the last report. This sample used the platinum wire anode and achieved a gross current of 500  $\mu$ A. Optical examination of this sample prior to emission testing suggested a poor, non-uniform pin tip geometry. This characteristic was later verified during post emission examination in the SEM and traced to an improper composition of the final chemical etchant. Interestingly no evidence of "melted" pins could be found in the area under the anode although the poor pin uniformity may have masked some damage accumulated during emission.

It was anticipated the next emission test sample would be a single or few pin emitter. As noted earlier in this report, the preliminary effort using the mechanical shaving technique in the SEM had shown the feasibility of achieving small number of pins, or perhaps even a single pin. However examination of the samples available after shaving in the SEM indicated the emission information from these samples would be inconclusive because of the extensive stubble present after the pins were shaved flush with the matrix. In many areas small jagged protrusions existed where the pins had been scraped off. As a consequence of this

geometry it was decided to simply rerun CBS Sample No. 8 (after repolishing and etching) using the small diameter wire anode and close down the interelectrode distance to minimize the emission contribution from peripheral pins outside the area covered by the anode.

Composite Sample No. 8 was reprocessed and installed in the test diode with an anode-cathode spacing of approximately 0.045 inches for the initiation of run CBS No. 9. This separation was intentionally left larger than the desired 10 mill gap because of the anticipated anode heating and subsequent expansion of this structure. After bake-out with heating tapes and pump-down to  $10^{-7}$  torr emission was initiated at 5 Kv and, as usual, the emission rapidly increased and with voltages of about 4 Kv a  $\mu$ A of gross current was obtained.

During the initial conditioning period of Sample No. 9 it was verified that the emission noise level was affected by the series resistor present in the diode circuit, the input resistance of the power supply, and voltage regulators used in conjunction with the power supplies. As suspected it is a very difficult task to separate changes in the real field emission characteristics from the small current variations associated with the electrical components. During a four day period the anode voltage was increased up to 9 Kv with a current increase to 200-300  $\mu$ A. The noise characteristics during this period remained typically 5-10% of the emitted current. During the fifth afternoon of testing the voltage was increased to 10 Kv and the sample was left overnight. These conditions

had been utilized for a short duration during the afternoon and it was anticipated these settings would further heat the anode and reduce the interelectrode spacing. Unfortunately during the night the ion pump tripped out and extensive vaporization and excessive current conditions were evident. The following morning the power supply remained at 10 Kv but the emission current was less than  $10 \mu\text{A}$  in a system pressure of  $10^{-4}$  torr. It is uncertain whether this failure was initiated because of a line voltage fluctuation which tripped the ion pump or resulted from a decreasing interelectrode spacing (increasing current) which either shorted or arced causing excessive vaporization which overloaded the ion pump. Post emission examination of both the anode and cathode from this experiment revealed that the anode, initially polished flat, had rounded and significantly decreased in area; whereas the cathode showed a small "pile" of metal supported on the pins. These results are not typical of experiments run with bulky anode assemblies where most of the damage is found in the cathode structure.

As a consequence of this difficulty this experiment (CBS - No. 9) could only duplicate the results found previously during CBS - No. 8, and field emission currents of  $\sim 500 \text{ nA/pin}$  from the oxide-metal cathode structures were experimentally duplicated.

#### IV. Financial Report

At the end of April the contract funds remaining were \$1200. These funds will be used for salaries, miscellaneous supplies and SEM

examination of several low-voltage structures. A contract extension through June 30, 1975 has been proposed.